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Chemical Characterization and Toxicological Evaluation of Airborne Mixtures; A System for Generating Mixed Aerosols from a Petroleum Based Liquid and a Fine Solid

FINAL REPORT

March, 1989

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Reduction of sound from the solids generator was accomplished and a system to remove the spent aerosols was assembled. Housings for safety, noise control, and a confinement of fugitive solids were designed for each aerosol system. The report presents the details of the system, with engineering drawings, and limited physical and chemical characterization of the material produced.

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EXECUTIVE SUMMARY

As an ongoing effort on the part of the U. S. Army to investigate the toxic potential of various materials employed as obscurants, a laboratory scale system is required to generate, in a controlled manner, mixed aerosols from a petroleum based liquid (PBL) and a candidate solid material. The aerosols are to mimic those generated by the field unit, which is capable of delivering several gallons of the PBL and several pounds of the solid per minute. The laboratory generator must deliver these components continuously for several hours at concentration levels of 0.1 mg/L - 5 mg/L. The aerosol so generated will be diluted in air (-500 L/min) and delivered into test chambers where animals will be contained for inhalation toxicological studies.

A system has been developed at Oak Ridge National Laboratory (ORNL) which is capable of delivering the mixed aerosol at the desired concentrations continuously for several hours. The PBL aerosol is generated by an evaporation/condensation process using a system similar to that previously developed at ORNL to generate diesel fuel aerosols. For the purpose of this investigation, the generator was modified to meet requirements for generating the PBL aerosol. For aerosolization, the solids are dispersed using a commercially available jet-mill system. A screw feeder delivers the solids at a constant rate into the jet mill where the particles are de-agglomerated and dispersed into a delivery stream of air. It was necessary to modify both the screw feeder and the jet mill to accommodate the candidate material. After generation, the two aerosols are blended for delivery into an exposure chamber.

Reduction of sound from the solids generator was accomplished and a system to remove the spent aerosols was assembled. Housings for safety, noise control, and confinement of fugitive solids were designed for each aerosol system. This report presents the details of the system, with engineering drawings, and limited physical and chemical characterization of the material produced.

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SYSTEM DEVELOPMENT

Petroleum Based Liquid Aerosol Generator

System Description

The PBL generator is a modification of a generator developed in this laboratory for generating an aerosol from diesel fuel (Holmberg, et al, 1982). The generator employs the evaporation/condensation technique for generating the aerosol. A schematic diagram of the PBL generator is portrayed in Figure 1. A one (1) kilowatt Vycor encased immersion heater is mounted in a 1 inch diameter stainless steel tube. The temperature of the heater is monitored by thermocouple and controlled at 1100° F, by a Barber-Coleman Temperature Controller. The PBL is pumped at a constant rate using a HPLC type metering pump onto the surface of the Vycor encased heater where it is flash evaporated. Nitrogen carrier gas sweeps the vapors out of the generator into a stream of dilution air where the vapors are cooled and condense to form the aerosol. Descriptions and detailed drawings of the system are provided in the appendices to this report (Figures A-1 - A-9).

PBL generator modifications

The original design (Holmberg, et al, 1982), employed a much longer generator tube (35 inches), with the downstream end of that tube being maintained at a temperature of 350° C using a heating tape. This was designed to provide an environment such that the aerosol would be generated under realistic conditions. The real smoke screen is generated by vaporization of diesel fuel in the exhaust manifold of a battle tank, with the vapors being carried with the exhaust into the air, where the vapors cool to form an aerosol of diesel fuel. The length of the laboratory generator tube was chosen so as to provide conditions which simulated the real exhaust temperatures of approximately 350° C.

Generation of an aerosol based on the PBL with the diesel fuel aerosol generator described previously at the temperatures used with the diesel fuel was found to be unsatisfactory. The exit temperature was too low and condensation of the PBL occurred in the exit nipple of the generator. Shortening the generator tube and eliminating the cooling zone caused the exit temperature to be much higher (-700° F), and no condensation at the exit was observed.

Nitrogen gas is used as carrier to prevent chemical reaction on the hot surface of the Vycor heater. It is considered unwise to use air as the mixture in the generator tube will be above the explosive range for hydrocarbons in air (> .50 mg/liter). The system was tested with up to 20% air blended into the carrier gas. No chemical changes were observed. In previous work, an aerosol of a petroleum based liquid made to earlier military specifications was generated with up to 50% air blended with the nitrogen carrier. In those tests some chemical transformation was observed (Holmberg, et al, 1982). No definitive identification procedure was used.

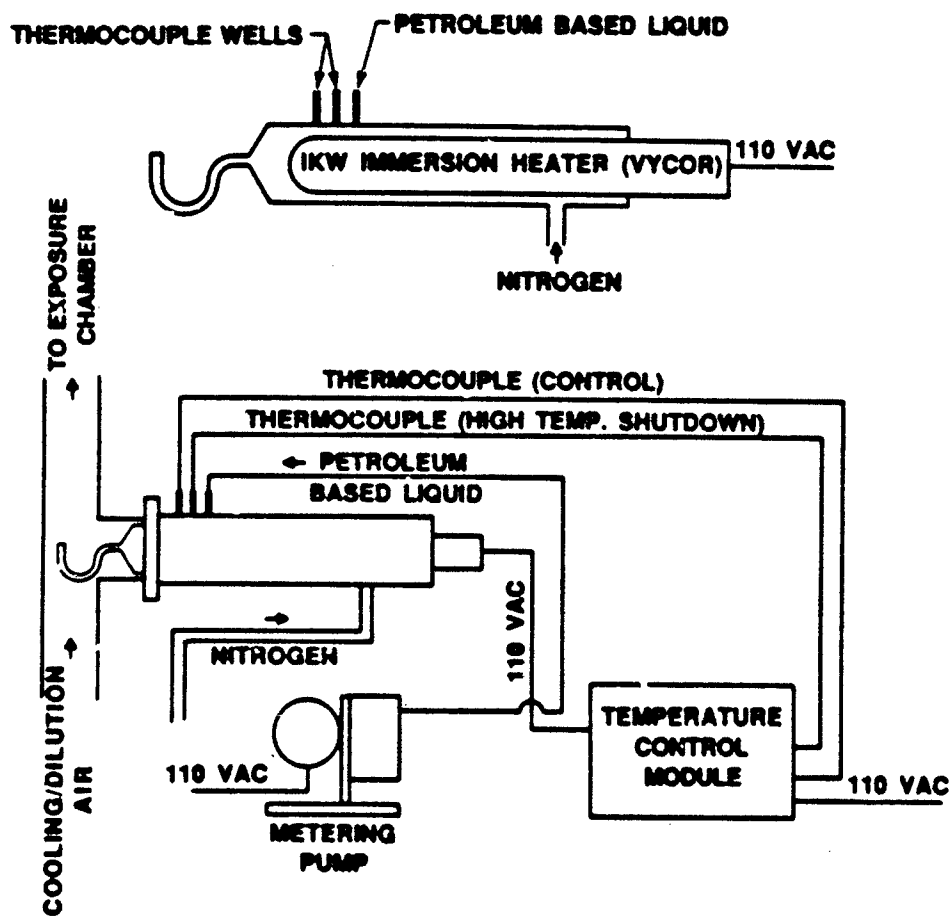


Figure 1. Petroleum Based Liquid Aerosol Generator

Solids Aerosol Generator - System Description

The solids dispersal system is a commercially available system modified at ORNL to more effectively deagglomerate the candidate solid material. The system consists of a small jet mill dispersing unit (Jet-O-Mizer Model 00, Fluid Energy Processing and Equipment Company) with solids supplied by a screw feeder (Series 100, AccuRate Inc.). The throughput of the system is controlled by the revolution rate of the feeder screw and the feed screw size. Solids, delivered by the screw feeder into the jet mill funnel, are drawn into the mill by aspiration and accelerated to high velocities by two air jets. The particles are swept into turbulent motion and pulverize each other. They are then swept into a particle size classifier and, if small enough, are carried from the jet mill into the delivery tube. Large particles are returned to the mill for further size reduction. Figure 2 is a schematic diagram of the solid dispersal unit. Figure 3 is a photograph of the combined systems showing the delivery tubes and a blending Y-tube at the entrance to the exposure chamber.

Screw feeder modifications

The screw feeder incorporates a flexible hopper designed to promote uniform delivery of the solid material by preventing it bridging over the delivery screw. A vinyl liner is used in the hopper and the flexing paddles are outside the liner. During early testing of the solid dispersal system with the candidate solid, it was found that the screw feeder did not deliver the material in a smooth and reliable fashion. The action of the flexing paddle in the hopper was not sufficient to prevent bridging of the solids over the screw and significant packing occurred between the screw and the delivery tube walls. Solids delivery to the jet mill was not uniform and would frequently stop altogether. The system was modified in order to achieve a more uniform delivery. A stirrer that moves up and down over the feeder screw was fabricated from a 1/8 inch diameter stainless steel welding rod. The stirrer, hinged at the front of the screw feeder, is agitated by the paddle in the feeder hopper. A small air driven vibrator is attached to the stirrer. The stirrer prevents bridging over the screw, and the solid material is readily collected by the screw. Even with this modification, it is necessary to periodically push the solids down from the hopper walls manually and to maintain the level of solid material in the hopper (approximately 1/2 full) in order to obtain constant delivery.

As the solid is drawn by the screw through the delivery tube, significant packing occurs, and the feed rate becomes variable. To obviate this problem, a second vibrator was attached to the delivery tube from the feeder. This vibrator helps to prevent packing in the delivery tube. With these modifications, delivery rates vary by only a few percent. (See the calibration curves [Figures A14, A15, A16] for the screw feeder in the Appendix)

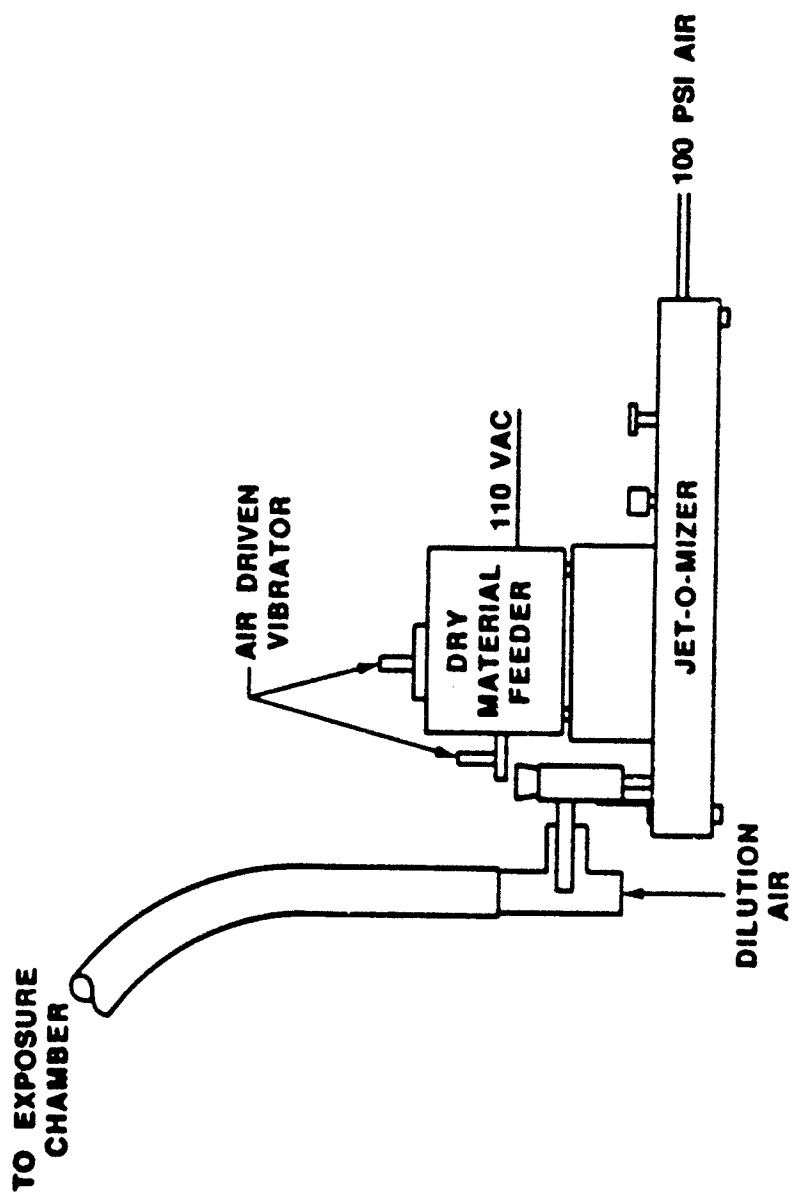


Figure 2 Schematic Drawing of the Solid Dispersal System

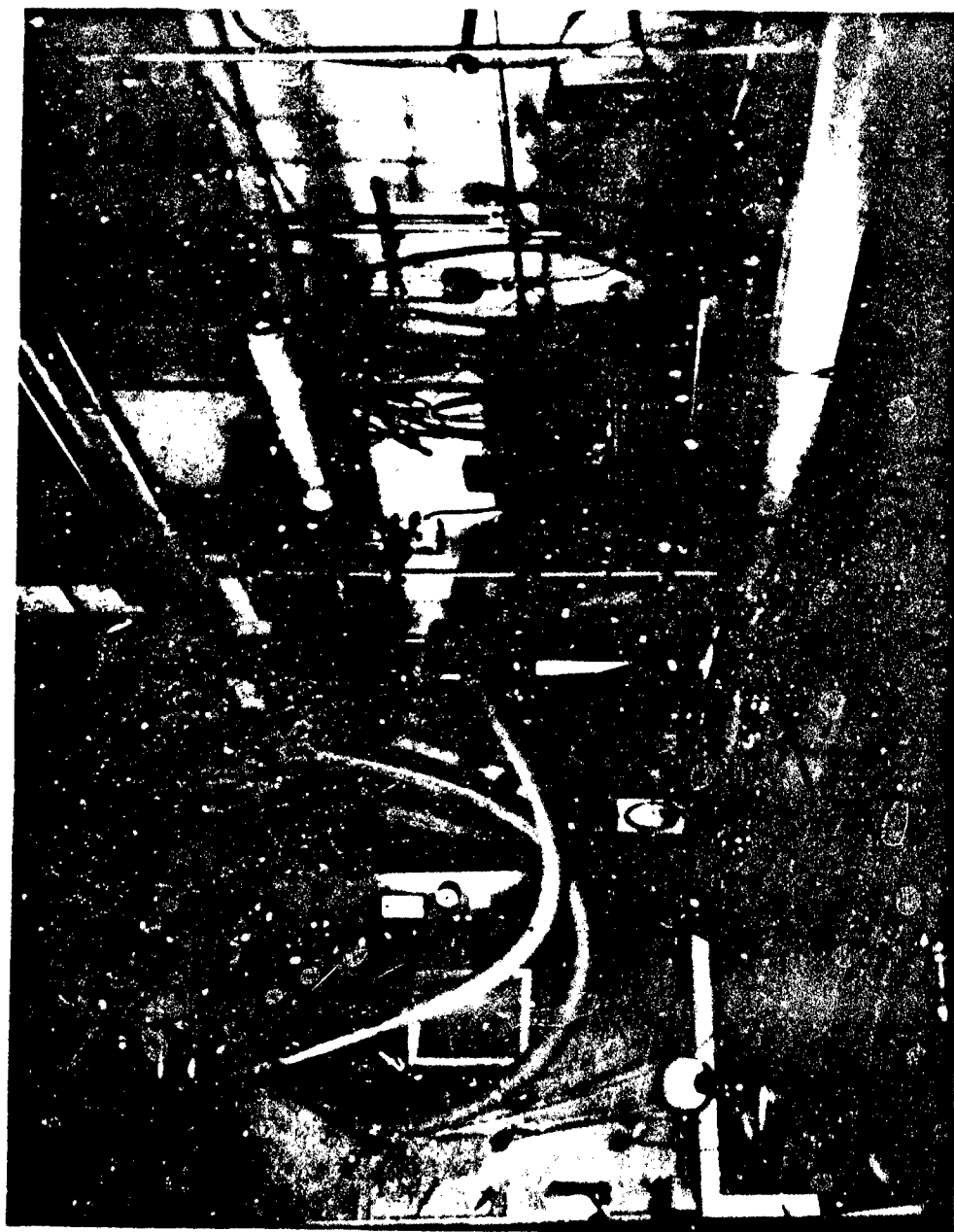


Figure 3 Combined PBL/Solids Aerosol Generator System

Solid Dispersal Unit Modifications

Some large particles are either emitted from the jet mill or are formed in the delivery tube. To prevent these particles from entering the exposure chamber, an extension of the jet mill delivery tube was added in order to impinge the particle stream against the plenum wall prior to exiting the system. Large particles impact against the wall and soon fall to the bottom of the plenum. A side arm is provided at the bottom to collect these solids and prevent them being swept further into the exposure system.

Occasionally, solids dispensed into the funnel of the jet mill were found to collect on the upper wall of the funnel. This is probably due to accumulated static charges on the solids. At times, this accumulation was so great that built-up solid material would fall off the outside of the funnel and be lost. A shroud or funnel extension was fabricated from heat shrinkable tubing around and above the mouth of the funnel. The delivery tube from the screw feeder is inserted through a hole in the side of the funnel extension. The aspirator drawing the solids into the mill pulls sufficient air down the funnel to prevent the accumulation of the solids on the funnel wall extension.

Sound control

Air at a pressure of 100 pounds per square inch (psi) is used to operate the jet mill. An air driven aspirator draws solids from the inlet funnel into the mill. The aspirator emits a particularly loud high frequency sound. Additional air jet noise is emitted from the jet mill through the delivery tube from the mill. Much of this noise is transmitted to the exposure chamber and to the area in which the human operator of the system may be standing. Prior to noise abatement efforts, sound levels were greater than 90 decibels at the face of the generator and in the exposure chamber.

Noise abatement was achieved in part indirectly through a modification of the system intended for another purpose. Extending the delivery tube of the Jet-O-Mizer in order to more efficiently cause impaction of the larger solid particles against the wall of the glass plenum also reduced some of the noise present in the exposure chamber. Initial attempts to extend the delivery tube using glass or stainless steel increased the noise level in the chamber. By fabricating the extension from heat shrinkable tubing, molded around a tube of the same diameter as the inside diameter of the original delivery tube, the noise level was reduced to approximately 80 decibels in the exposure chamber. However, this had little effect upon the noise level experienced by the operator. Most of that noise originates at the jet mill inlet funnel and aspirator. Therefore, a wooden housing with a transparent cast acrylic cover was constructed to enclose the entire assembly. This housing not only reduces the external noise level, but helps to confine fugitive solid particulates and permits ready observation of the conditions in the feeder and the funnel of the Jet-O-Mizer. Figure 4 shows the solids dispersal unit mounted in the housing, as well as the stirring bar, vibrators and funnel shroud.



Figure 4 Solid Dispersal System Mounted in the Housing

Spent Aerosol Removal

Neither the solid nor PBL aerosols should be vented directly into a conventional laboratory hood system. The two aerosols, together, form a rather intractable mass. Filtration, or other particulate removal systems with which we had prior experience, were evaluated, but proved to be inadequate for this aerosol. In general, these systems loaded too rapidly to permit sufficient operating time to fully investigate the generating system.

A filtering system was finally devised which removed the aerosols effectively and yet has the capacity to permit extended runs. The system consists of a bag type "roughing" filter, followed by a high efficiency filter. The roughing filter is a Cambridge Model 3Z95 fiberglass filter. The filter consists of five (5) bags or envelopes mounted in parallel configuration. The filter overall is 12"x 24"x 29", with the opening of the five bags on the 12" x 24" side. The configuration yields a filtering surface area of 48 square feet and is rated at 93-97% efficiency against atmospheric dust and has a holding capacity of 30 grams/square foot. This is equivalent to 1.44 kg (3.2 pounds). One filter used in the developmental work was loaded with over 6 pounds of the aerosol materials before the pressure drop across the filter became too high to maintain proper air flow. The filter for the developmental work was mounted in a plywood housing. Figure 5 shows the open filter housing with a used filter in place. A clean filter is shown to the right of the filter housing.

The backup filter is also manufactured by Cambridge (Type CM113A). This filter material is rated at 99.99% efficiency for particles of 0.3 μ m diameter and is the same material used for collecting analytical samples from the aerosol generating system. A 1.5 square foot section is mounted in a plywood housing with a 1/4 inch mesh stainless screen filter support. Typically, no particulates were detected downstream of this filter. Figure 6 shows this final filter system in a disassembled configuration. The magnehelic gauge in this figure is used to monitor the pressure drop across the secondary filter. By disconnecting the upstream hose, the pressure drop across the entire system may be determined. This is useful in determining the loading of the filters.

This filtering system permits operation of the generating system for extended periods of time without shutdown. As the filters load and the pressure drop increases, adjustments in the degree of suction applied to the overall system must be made to maintain correct airflow. However, loading is slow enough that only minor adjustments are necessary, even during runs as long as four hours. The cost of the filters is sufficiently small (\$35 each) that even daily changes are not prohibitively expensive. Presumably, other high efficiency filters with greater surface areas than that used here as a backup filter would permit longer operating times.

It must be emphasized that spent aerosol removal is a significant problem with this material. The blended aerosol will completely seal the surface of a filter material, so that air flow is essentially stopped. Experience has shown this to occur rapidly with some of the filtering systems tested. The danger is that as air flow is restricted, the concentration

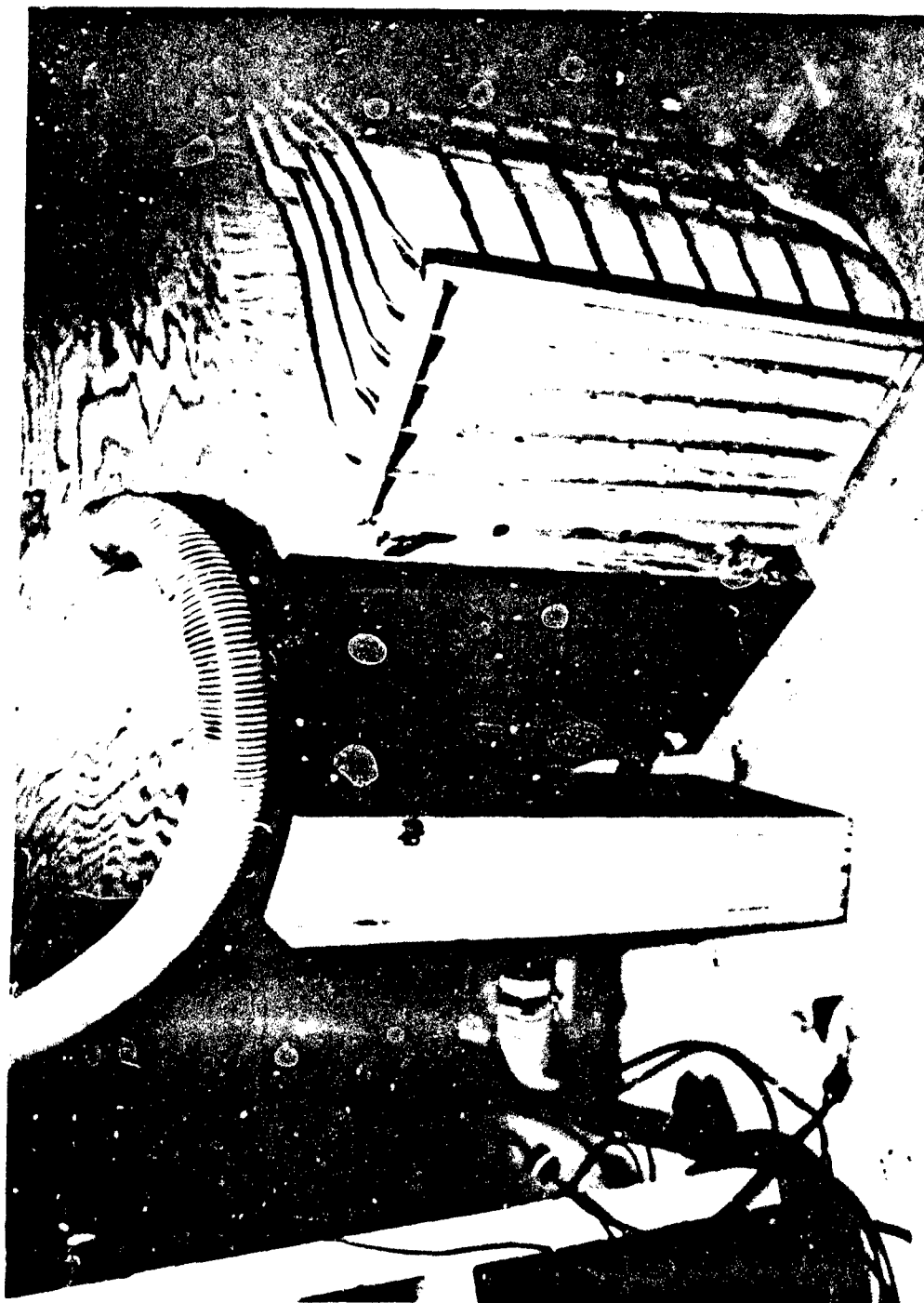


Figure 5. Spent Aerosol Removal System; Pre-filter and Housing

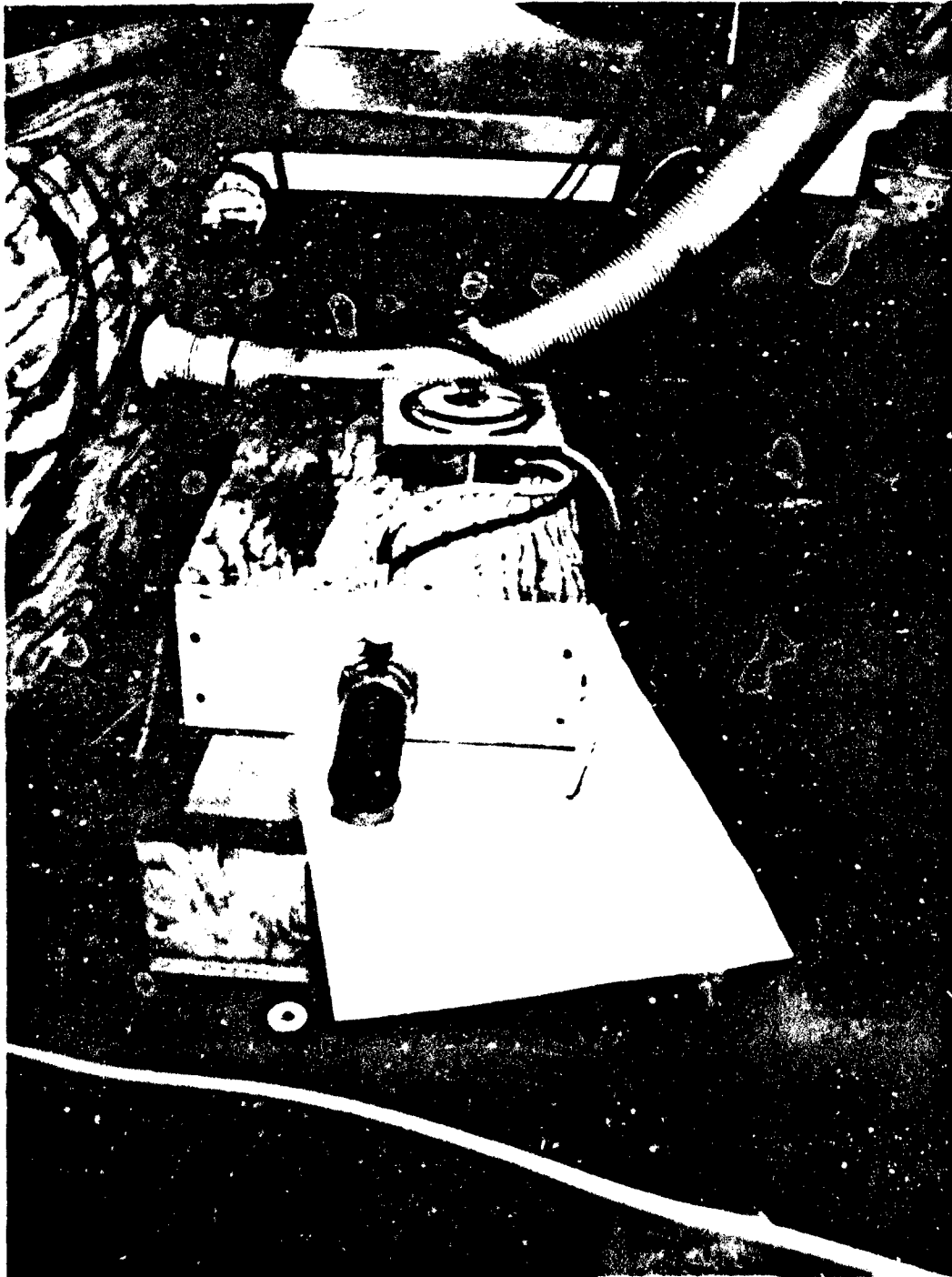


Figure 6 Spent Aerosol Removal System: Final Filter

of both aerosols increases to the point where the PBL aerosol could reach an explosive level. Although the PBL aerosol was not tested for its explosive limit, the limit is expected to be approximately equal to that of the diesel fuel aerosol investigated previously. In that study, fuel based aerosol was generated by the evaporation/condensation technique. The concentration for these tests was calculated from dilution air flow rate and diesel fuel delivery rate from the metering pump into the aerosol generator. To prevent collecting an explosive mixture in a chamber, the delivery tube from the generator was vented into the exhaust from a laboratory hood where the aerosol was rapidly diluted to a safe concentration. Dilution air was delivered by positive flow rather than being drawn through the system as described in this report. Because it has not been reported previously, the procedure for determining the explosive limit of diesel fuel aerosol is reported here, along with the results.

A 300 mL test bomb was prepared by capping the ends of a 2 inch PVC pipe. One end was fitted with an open collecting tube (1/4 inch I.D.) and the other end was fitted with a 1/4 inch tube and a toggle valve. A sample could be drawn into the bomb by vacuum at the toggle valve and sealed by plugging the open tube with a stopper and closing the toggle valve. Two electrodes were mounted in the sides of the bomb. One electrode was connected to ground and a Tesla coil applied to the second electrode generated an electrical arc through the aerosol. Samples were collected from the delivery tube, the toggle valve closed, the open collection tube sealed and the system removed from the vicinity of the delivery tube. An electrical arc was then passed through the aerosol.

At a diesel aerosol concentration of ~60 mg/L (includes the diesel aerosol and vapor) only 1 in 12 tests was positive (an explosion occurred). Above 60 mg/liter concentration increasing at 1 mg/liter steps, over 50% of the tests were positive. At 65 mg/liter 7 of 11 tests were positive. Although the technique was crude, the values are in reasonable agreement with the literature values of explosive limits for other hydrocarbons, (hexane - 45.3 mg/liter, octane - 50.0 mg/liter and decane - 49 mg/liter as calculated from literature data (West, 1965).

It is recommended that the exposure chambers be equipped with flow monitors that will sound an alarm if air flow drops below a redetermined level. Operation at concentration levels at or below 5 mg/L maintains a safety factor of ten.

AEROSOL CHARACTERIZATION

Physical Characterization

Experimental

The system has been evaluated in three modes of operation; PBL aerosol only, solid aerosol only, and the aerosol formed from the blending of the two. In general, for the blended aerosol, the two systems have been operated at approximately equal aerosol concentrations.

Mass Concentration Monitoring

During operation with the liquid-based aerosol, concentration can be monitored by using the ORNL infrared backscattering detector (Higgins, et al, 1978). When operating the solids generator, either alone or in a blended mode, the optical detector is ineffective, since the solids rapidly coat all surfaces, including the detector. This diminishes both the outgoing and backscattered beam to the point that the detector becomes opaque. The concentration of either the solids-only or the blended aerosol was determined by collection of aerosol samples on filters and subsequent weighing. To determine the mass concentration of the individual components of the blended aerosol, the PBL is extracted from the solids on which it is absorbed using iso-octane. Any solid material remaining in the extract is removed by filtration, and the concentration of the PBL in the resulting solution is determined by measuring the absorbance in a UV spectrophotometer at 255 nm, and comparing with standard solutions of the PBL.

Particle Size Determination

The distribution of aerosol particle sizes has been determined by cascade impactor measurements (Mercer, et al, 1962). Seven stage impactors of the Mercer/Lovelace design were used. The impactors are operated at a flow rate of 1 liter/minute, collecting the particulates on stainless steel substrates. When sampling aerosols composed only of solids, the substrates were coated with a light film of Apiezon M grease to prevent particle bounce. Stage loadings for the single PBL aerosol were dissolved in iso-octane measured by UV spectroscopy at 255 nm. Solid-only and blended aerosol loadings were measured gravimetrically.

Data reduction for the impactor measurements has been described in detail elsewhere. (Jenkins, et al, 1983). Briefly, cumulative mass percent loadings for individual impactor stages (mass of the particles having diameters less than the cut off diameter of that stage) are plotted using normal probability coordinates vs. the logarithm of the diameter. Although the blended aerosols did not yield a linear fit, a log normal distribution was assumed and the best straight line was drawn. Figure 7 is a graphical representation of the distribution of blended aerosol, while Figures 8 and 9 are graphs of similar distributions for the PBL-only and the solids-only aerosols.

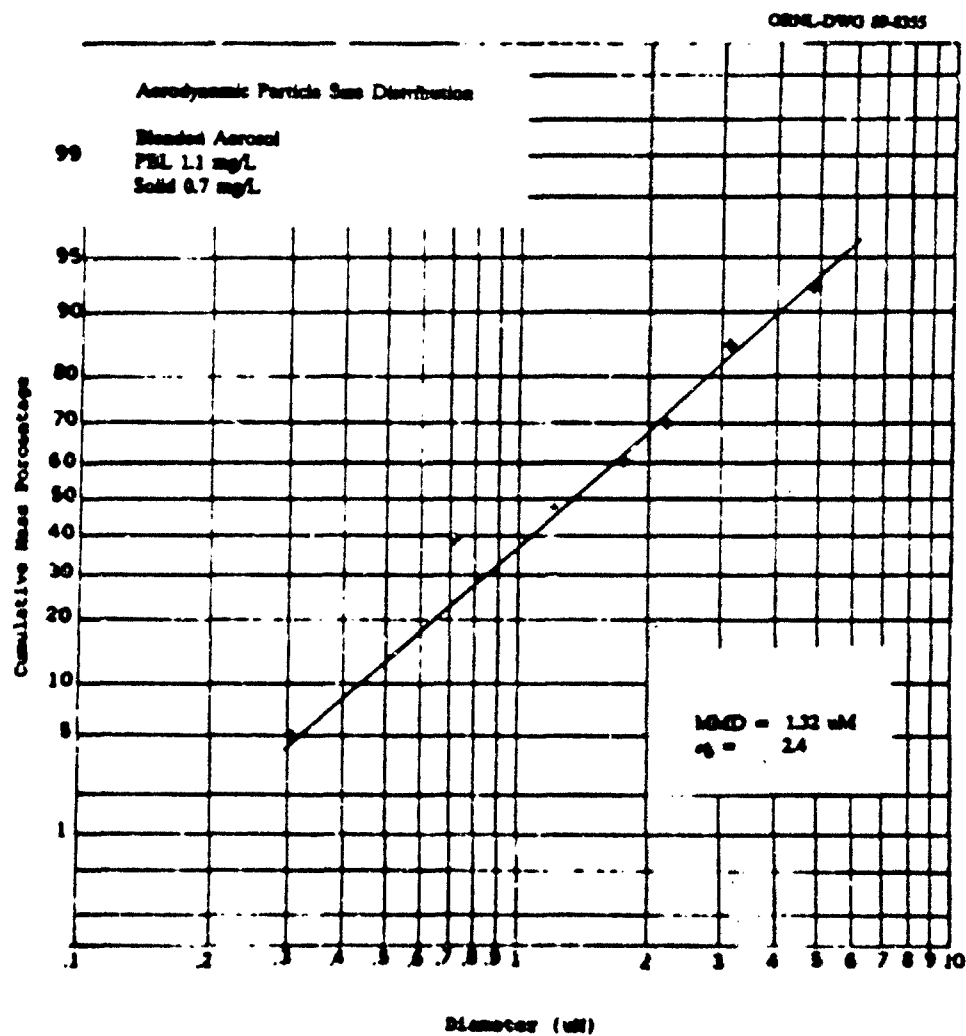


Figure 7. Particle Size Distribution; Blended Aerosol

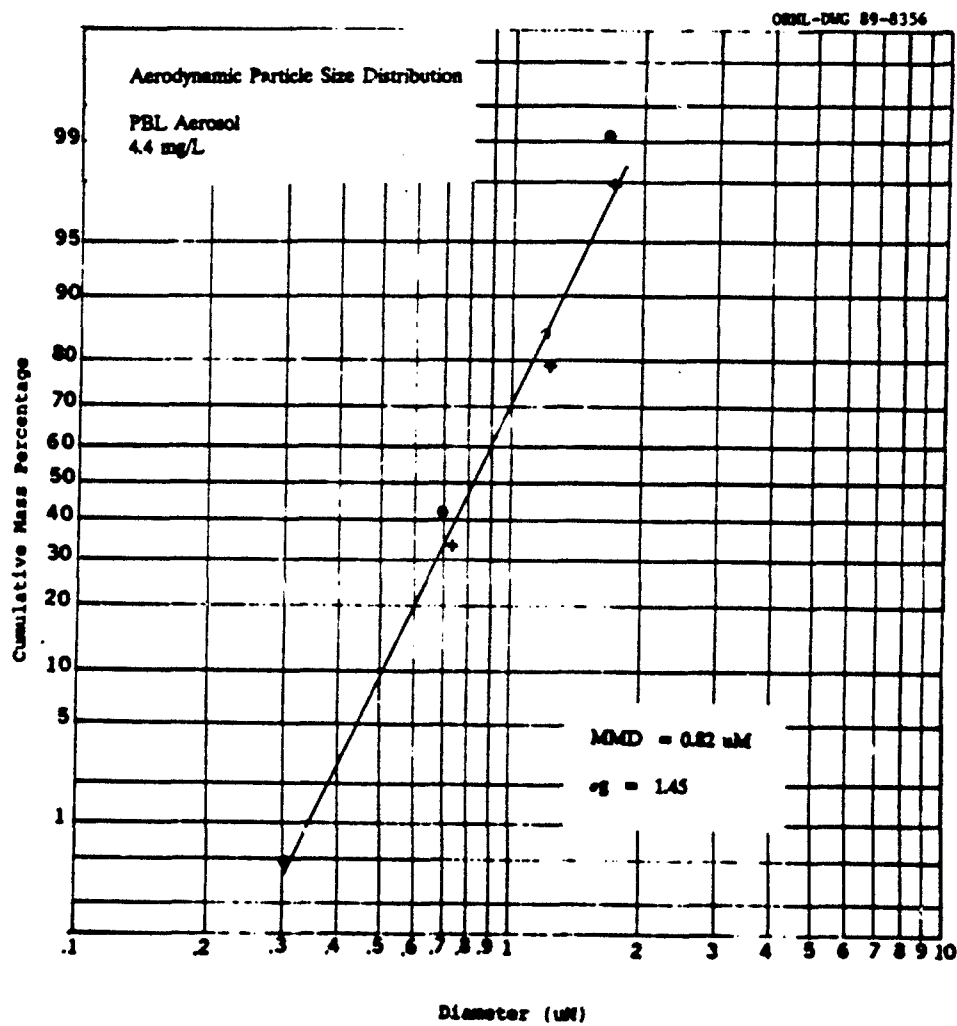


Figure 8. Particle Size Distribution; PBL Aerosol

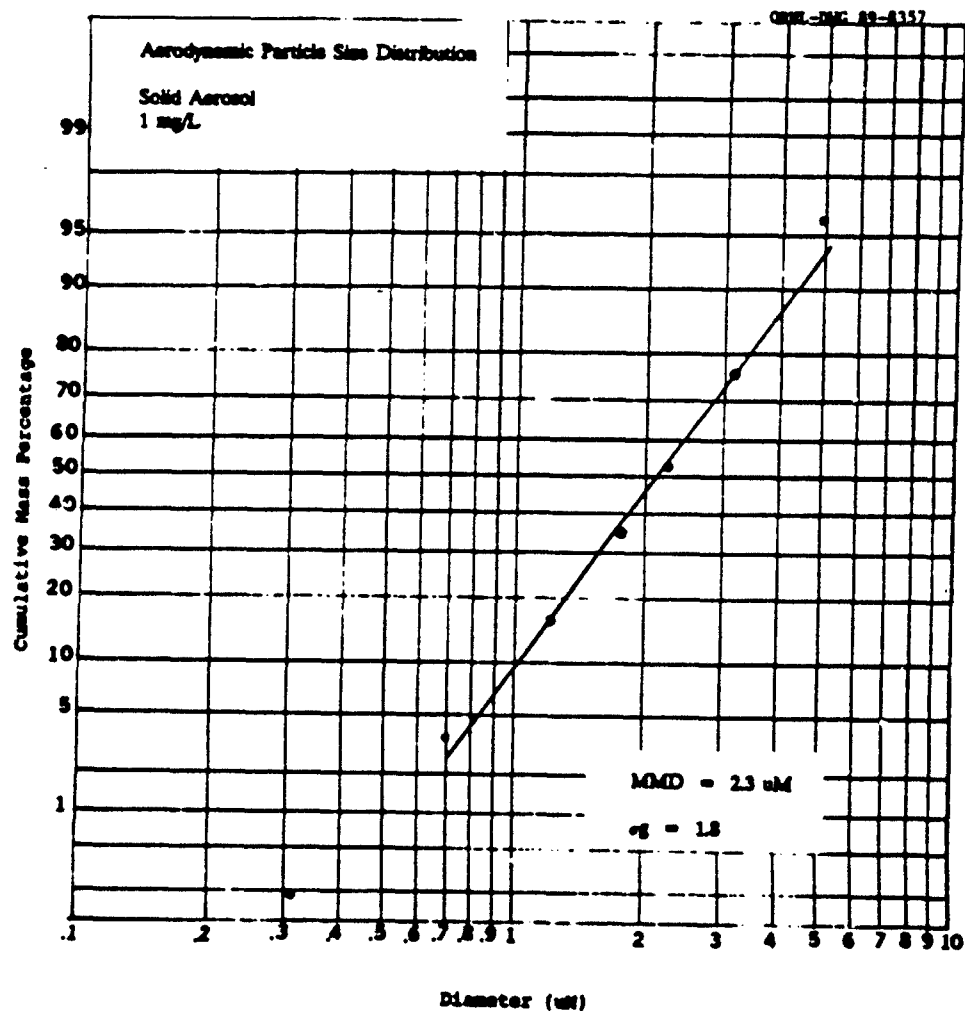


Figure 9. Particle Size Distribution; Solids Aerosol

To determine the relative mass of each of the components in the blended aerosols, the individual stages were leached with iso-octane. To separate the solids, a portion of the solution was forced through a disposable filter assembly (Gelman ACRO LC13 0.45 μm). The absorbance of the resulting clear solution was then determined by measuring at absorbance 255 nm as described above. Although a log normal plot of the blended aerosol impactor stage data does not yield a straight line fit, a log normal distribution was assumed and the best straight line was drawn.

Chemical Characterization

Methods

Limited chemical characterization studies of the PBL-only aerosol were performed. In order to determine if gross changes had occurred in the PBL as a result of aerosolization, spectrophotometric observations were made of the UV absorption region for both the aerosolized and un-aerosolized PBL. Samples of the extracts were then analyzed by high pressure liquid chromatography (HPLC). For this analysis, an Alltech Spherisorb NH_2 (5 μm) column (250 x 4.6 mm ID) and a Beckman HPLC system were employed. A refractive index detector coupled in series with a fixed wavelength UV detector (254 nm) was used to monitor the effluent. Hexane and hexane/methylene chloride were used as the mobile phase at a flow rate of 1 mL/min. Fixed volume injections (20 μL) of the sample were made with a Rheodyne loop injector. Initially, all samples were analyzed using a step gradient: 100 % C_6H_{14} for 18 min followed by 100% $\text{C}_6\text{H}_{14}/\text{CH}_2\text{Cl}_2$ (1:1) for an additional 17 min. It was determined, however, that perturbations in the baseline of the UV detector, under these conditions, would preclude accurate measurements of more polar constituents eluting with the front of the polar solvent. Therefore, some analyses were performed using a linear gradient: 5 min. at 100% C_6H_{14} followed by a shift to 100% $\text{C}_6\text{H}_{14}/\text{CH}_2\text{Cl}_2$ (1:1) over an 18 minute interval, with a 3 minute hold before returning to the original conditions.

Sample types of PBL collected and analyzed by this technique included aerosolized PBL, fresh PBL (not aerosolized), and a comparison of PBL aerosol generated using only nitrogen carrier and PBL generated using a nitrogen carrier with up to 20% air (4% oxygen) added to the carrier. Extracts of the filters (Cambridge CM113A) used to collect the samples did not yield sufficient background to interfere with the analysis. (The Cambridge filter was employed because of its known retention of aerosolized materials and our experience with the filter material.)

Results and Discussion

Although one of the purposes of the generation system is to produce the combined aerosols of the PBL and the solid, much of the characterization and effort has been directed toward the individual aerosols. Both aerosols are formed prior to blending, and there is some coagulation of particulates after blending. However, there is evidence that the PBL and the solid remain, for the most part, separate particulates, (see the discussion

on particle size distribution below). Although initially separate phases, the two phases do combine on filter surfaces and very quickly seal the surface of most filters and effectively stop air flow. For this reason, care must be taken to adjust the sampling time and flow rate so that the quantity of sample collected by filtration for analysis is not sufficient to alter the initial sampling rate. With aerosols of the blended material at approximately one to one ratio by weight, samples of up to approximately 40 mg have been collected on 45 mm diameter filters (~ 2.5 mg/cm²) without changing the sampling flow rates.

Petroleum Based Liquid

The petroleum based liquid aerosol is physically very similar to that produced by the diesel fuel aerosol generator described elsewhere (Holmberg 1982). It is an oily liquid, for which a small fraction of the lower molecular weight compounds do not recondense upon cooling and thus remain in the vapor state. Based upon comparison of the delivery rate of the metering pump and the final concentration of the aerosol, between 85-90 % of the PBL fed to the generator is delivered to the exposure chamber. There is some uncertainty in this measurement due to inaccuracies in air flow measurements. Very little of the PBL seems to be lost to the walls of the system.

When generated alone, the aerodynamic mass median particle size of the PBL aerosol is less than 1 μ m, with a geometric standard deviation of approximately 1.4. The particle size varies somewhat with mass concentration and ranges from 0.32 μ m to 0.8 μ m over the concentration range from 0.1 mg/L to 4.4 mg/L. This distribution is well within the respirable range. A summary of particle sizes as a function of mass concentration is given in Table 1.

No changes in the chemical composition of the aerosol from that of the starting material were detected. Although lower molecular weight compounds remain in the vapor state, the UV absorption studies made do not reflect this change. In Figure 10 are compared typical HPLC profiles for A (PBL not aerosolized), B (PBL aerosolized using 100% N₂ carrier), and C (PBL aerosolized with 20 % air added to the N₂ carrier). The profiles are very similar except for total concentration differences. Small differences observed in resolution and retention time are those normally associated with sampling, sample preparation and analytical variances. Elution profiles of other samples were similar. These studies, although far from exhaustive, suggest that no significant quantities of new chemical species were formed as a result of aerosolization through evaporation and condensation.

The aerosol concentration in the exposure chamber is a function of fuel pump rate and the dilution air flow rate. A delivery rate calibration curve for the prototype generator system is shown in Figure 11. The calibration curve for each system will be similar, but may be sufficiently different so as to require a separate calibration. The control vernier for each of the Eldex HPLC delivery pumps were found to be mounted slightly differently, thus requiring separate calibrations.

TABLE 1. PARTICLE SIZE DISTRIBUTION - PETROLEUM BASED LIQUID, SOLID AND COMBINED AEROSOLS

Aerosol Concentration mg/L	PBL		Solid Phase		Combined	
	MMAD*, μm	og	MMAD, μm	og	MMAD, μm	og
0.089	0.32	1.6	-	-	-	-
0.36	-	-	2.2	2.0	-	-
0.36	-	-	2.6	1.7	-	-
0.48	-	-	2.2	1.9	-	-
0.60	0.49	1.4	2.6	1.8	-	-
1.0	0.57	1.4	2.3	1.8	-	-
1.0	0.54	1.4	2.0	1.9	-	-
1.1	-	-	2.5	1.7	-	-
1.25	0.60	1.8	-	-	-	-
1.5 ^a	-	-	-	-	0.86	2.1
1.8 ^b	-	-	-	-	1.0	2.5
1.8 ^b	-	-	-	-	1.3	2.4
1.82 ^c	-	-	-	-	1.0	2.2
1.9	0.60	1.5	-	-	-	-
1.95	-	-	3.1	1.7	-	-
2.02	0.64	1.5	-	-	-	-
2.22	-	-	2.8	1.7	-	-
3.68	-	-	-	-	-	-
3.85 ^d	-	-	-	-	1.4	2.5
4.06	-	-	2.95	1.7	1.35	2.9
4.4	0.82	1.45	-	-	-	-

* Mass Median Aerodynamic Diameter

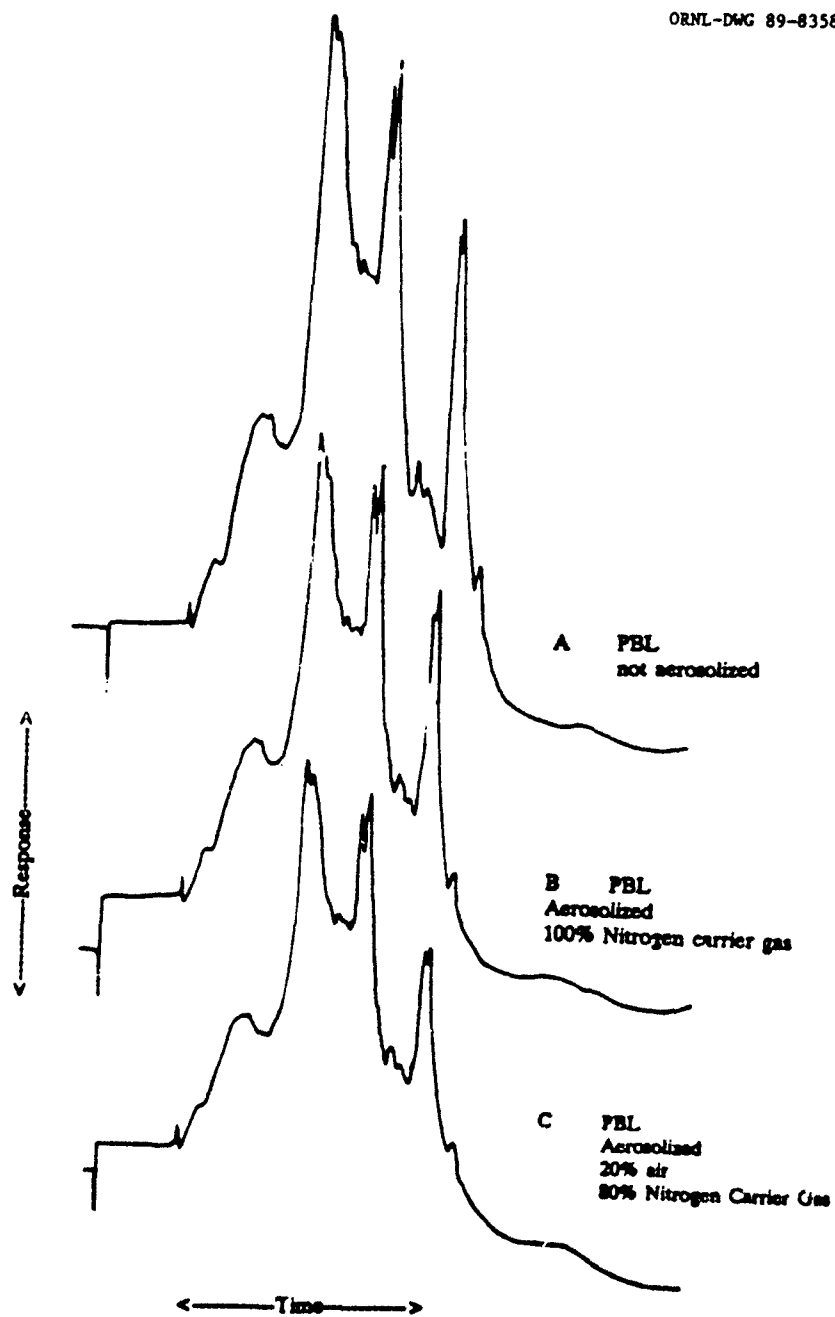


Figure 10. Typical Profile of High Pressure Liquid Chromatographic Analyses

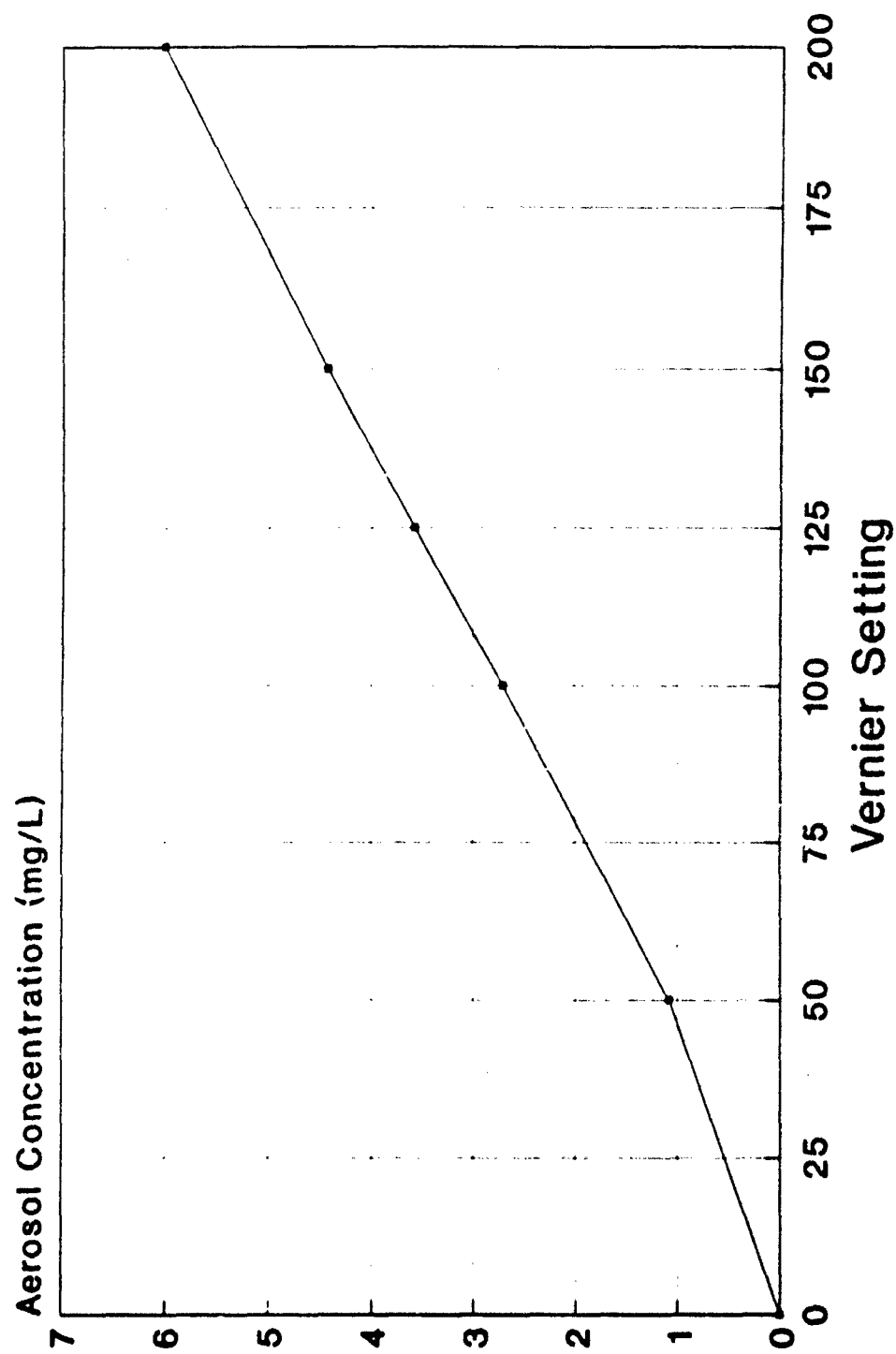


Figure 11. Calibration of Eldex Pump for Petroleum Based Liquid Aerosol Generator

Solids Aerosol

As only mechanical stresses are applied to the solids for the dispersement, no chemical assessment of compositional change was made, other than to determine that nothing that would interfere with PBL UV absorption measurements was removed by the iso-octane solvent from the solids. Similarly, the solid material was tested so as to determine its potential interference with the high pressure liquid chromatographic analysis. No interference was found.

Physically, the starting material has been described by the manufacturer as having a .5 to .6 μm particle size. These particles tend to agglomerate when brought together, and they are then partially de-agglomerated in the jet mill. Thus, the mass median aerodynamic particle diameter in the chamber was determined to be greater than 2 μm . At mass concentrations between 1 mg/L and 4 mg/L, the particle size of the solids-only aerosol ranged from 2.2 to nearly 3 μm mass median diameter (see Table 1). The starting material is, in reality, platelets of sub-micron thickness but may be larger in width or length. These particles will not behave aerodynamically as the spherical PBL particles. Although the density of the material is greater than 2 g/cc, the platelets or agglomerates of the platelets behave differently than spherical particles of that density. However, the density of the agglomerates is presumably much less than the density of the individual particles.

During the generation, the particles apparently acquire a significant static charge which affects their behavior. All surfaces are rapidly coated, and feather-like agglomerations grow from sites on surfaces in the exposure system and chambers. These agglomerates may be as long as 1/8 to 1/4 inch. Occasionally, they break loose from the growth site and have been seen to float through the exposure chamber. Some of these agglomerates were grown on slide cover plates and examined by electron microscopy. Figure 12 is a photomicrograph of one of these agglomerates. The agglomeration appears to be a random configuration. Some of the individual particles are observed to be larger than 1 μm . The concentration of these large agglomerations does not reach a level to significantly affect the particle size distribution. It is possible that these loosely bound particles would be de-agglomerated in passing through the impactor orifices and thus not affect the cascade impactor measurements.

Blended Aerosols

As stated previously, the two aerosols are fully formed before blending. Some coagulation of the solid and the liquid particulates does occur, but analysis of individual stages of the cascade impactor samples of the blended material indicates that the two materials remain, for the most part, separate aerosols. The cascade impactor data for the blended aerosol and for its solid and liquid components are plotted in Figure 13. The weight changes of the impactor substrates were used to determine the mass accumulation of the blended aerosol on each stage of the impactor. The mass loading of PBL for each stage was determined by extracting each stage with iso-octane and measuring UV

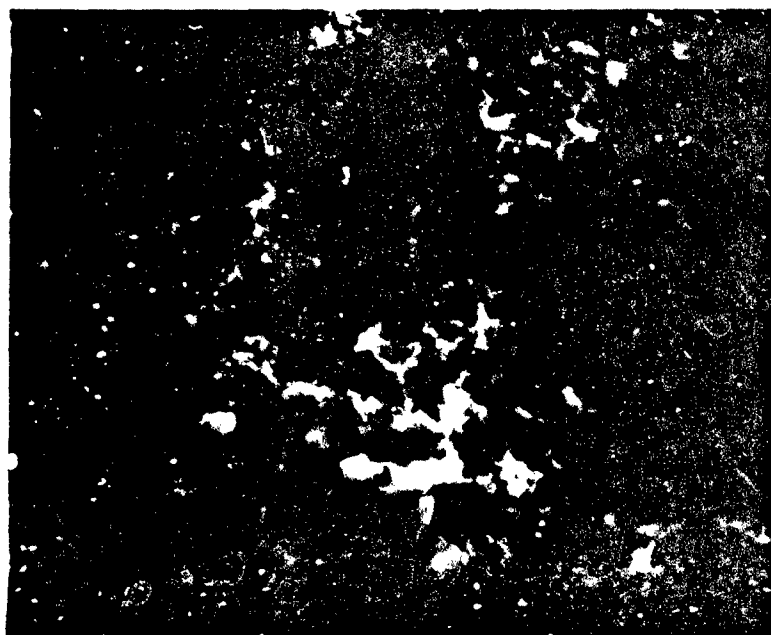


Figure 12 Solids Particulate Agglomeration; Scanning Electron Microscopy

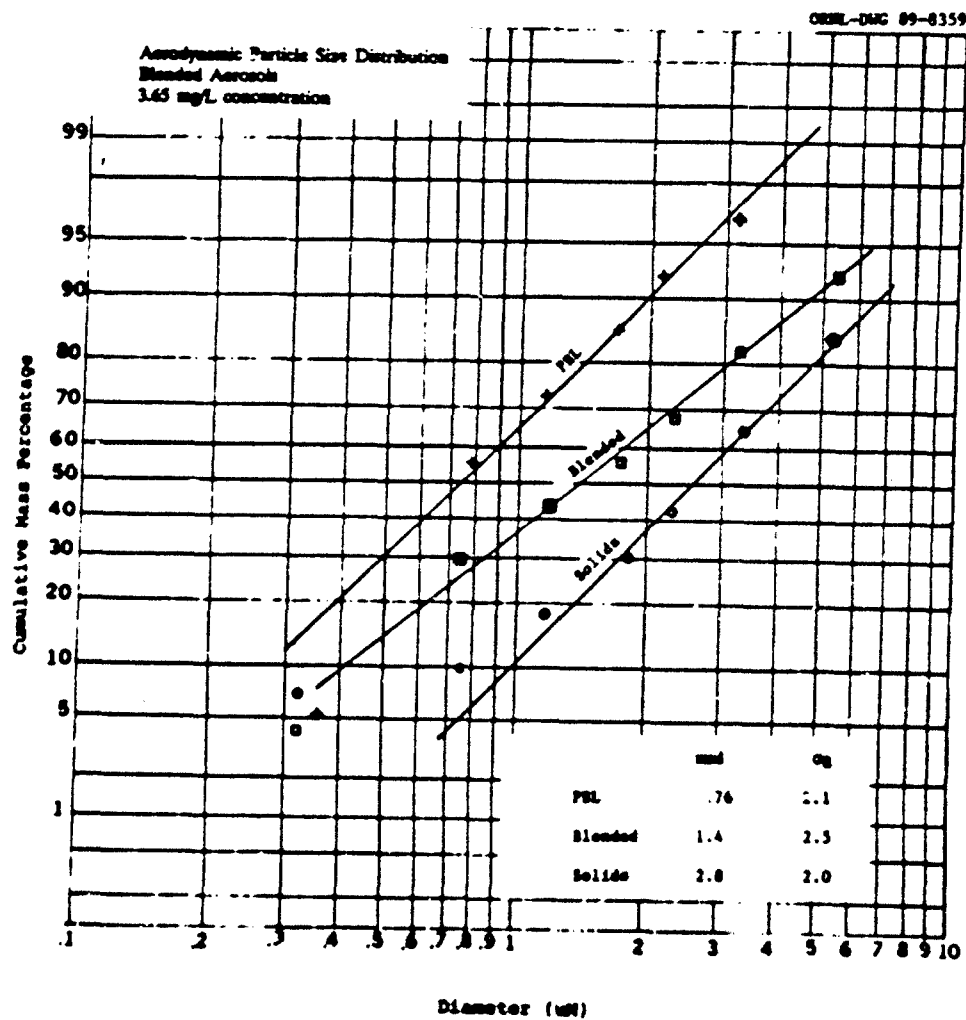


Figure 13. Particle Size Distribution; Blended Aerosol

absorbance at 255 nm. The mass loading of the solids was determined as the difference between the gravimetric and the UV absorption determinations. The data indicate that the individual aerosols are distributed log-normally, and that the apparent particle size distribution of the combined aerosol is approximately midway between that of the individual components. Little PBL aerosol is detected on the upstream stages (larger particle sizes), which contain most of the solids. Very little of the solids pass to the downstream stages that contain most of the PBL particles. The data in Figure 13 indicate that ca. 80% of the PBL aerosol is below the mass median diameter of the blended aerosols, while ca. 80% of the solids are larger. Thus, it appears that little agglomeration occurs between the two aerosols, and, as a result, the apparent aerosol particle size distribution of the blended aerosol is bimodal. In Figure 14 are depicted the particle size distributions of the two individual aerosols generated to form the blended aerosol whose distribution is portrayed in Figure 13. Comparing the particle sizes of each of the aerosols as determined when generated singly with that of the aerosol as determined from Figure 14, the data indicate that blending alters the mean aerodynamic particle size very little. However, the geometric standard deviation (indicative of the range over which the particles are distributed) is significantly greater. The larger geometric standard deviation is indicative of some coagulation occurring between the individual components of the blended aerosol. The mass median aerodynamic diameter of the blend is dependent upon the concentration of the individual components, and lies between the mass median aerodynamic diameters of the individual components.

It has been observed that the concentration of the blended aerosol does not equal the sum of the individual aerosols measured separately. For example, the concentration of the blended aerosol depicted in Figure 13 was determined by filtration to be 3.65 mg/L. The PBL aerosol generated and measured separately was 2.06 mg/L and the solid aerosol was 2.2 mg/L. This is an approximately 15% loss from the starting concentrations and cannot be explained by shift in delivery rate from the two generators. Presumably, the blended material is lost more readily to the walls than is either of the individual aerosols.

Wall Losses.

Loss of the aerosols to surfaces within the exposure system is a significant problem. All surfaces become coated with the materials and will create difficulties in cleaning the system after completing the daily exposure. An approach used during the development of the system was to vacuum the interior of the chamber using the exhaust line from the exposure chamber. Thus the same filter used to remove the particles from the spent aerosol was used to collect that material vacuumed from the walls. As stated previously, the bag filter is inexpensive (\$34 each) and could be changed daily if such a system is used in the exposure study. Other collection systems may prove to be more efficient and have greater capacities.

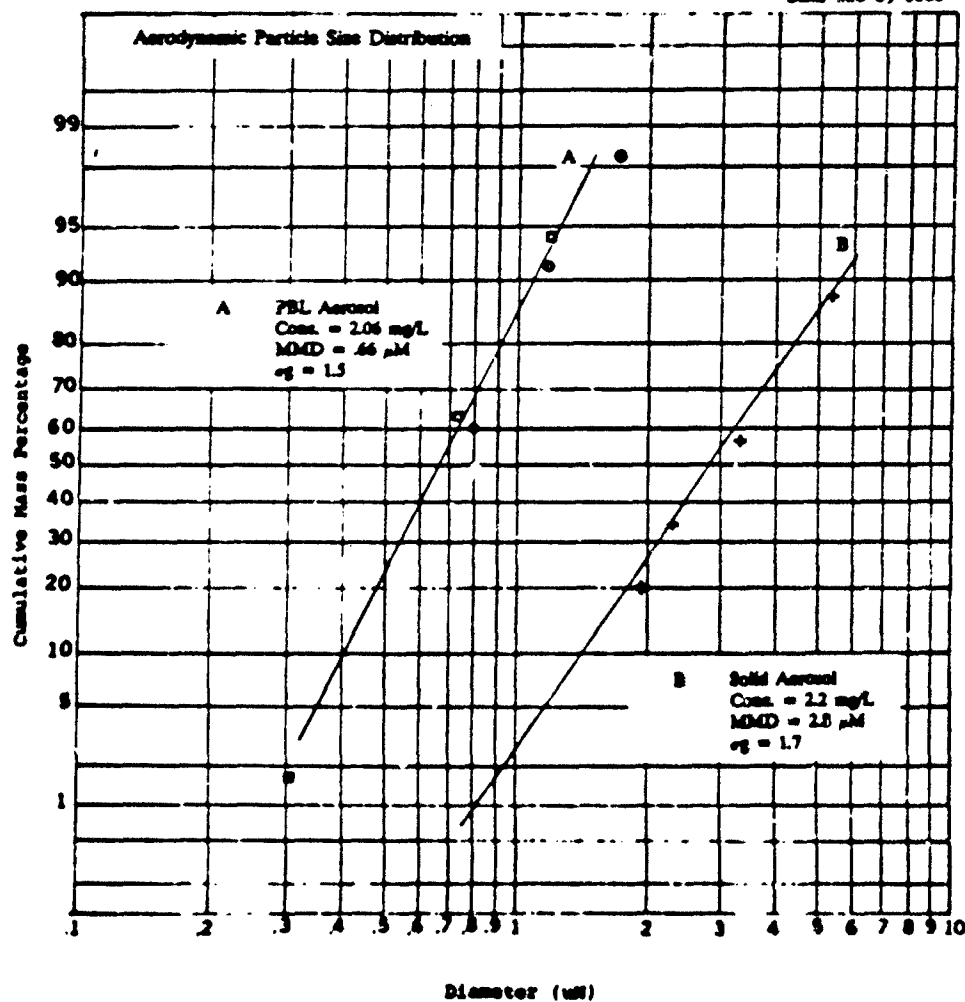


Figure 14. Particle Size Distribution: Unblended Solids and PBL Aerosols used to Generate the Blended Aerosol shown in Figure 13.

Aerosol Generation Stability

Constancy of the output of the generator during an exposure period is of prime consideration to the toxicologist. The concentration of aerosol to which the experimental animals are exposed must be documented. As discussed previously, light scattering techniques are not readily applicable to monitoring this aerosol mixture. To determine consistency of the output of the generators, filter samples were drawn from the chamber during extended runs. In Figures 15, 16, and 17 representative data are presented from runs of PBL-only, solid-only and blended aerosols, respectively. Figure 15 shows the data from a two hour run of PBL generated at a concentration of 1.43 mg/L. Filter samples of 10 minute duration were taken during the run and are plotted as individual points. The time for each data point is the midpoint in time of the sampling period from the starting time of the run. The mean value for nine (9) filter samples was 1.43 ± 0.04 mg/L. The dotted lines are arbitrarily chosen at $\pm 10\%$ of the mean value. All points lie well within the $\pm 10\%$ values.

Figure 16 presents similar data from a very low concentration solid aerosol run. Sampling periods for this run were extended to 30 minute periods in order to collect sufficient material to be statistically and analytically valid. For seven samples collected during the run, the mean value is 0.11 mg/L with a standard deviation of 0.01. Only one point of the seven lies outside the $\pm 10\%$ lines. In Figure 17 data are presented from a four hour duration run, with the blended aerosol at a concentration of 1.73 ± 0.06 mg/L. Sampling periods were 15 minutes each, and for 8 samples, none of the concentrations lies outside the $\pm 10\%$ lines. The concentrations of the individual aerosols blended for this particular experiment were 0.93 mg/liter for the solid and 0.90 mg/liter for the PBL. In this case, the concentration of the blend is only 95% of the sum of the two components. This apparent loss during blending has been observed for all studies with the blended systems. To determine the mass concentrations, filter samples were drawn from the aerosol in the exposure chamber as generated by the individual component generators. Generators would then be operated simultaneously and samples of the blended material collected. The loss is usually about 5% based upon these filter samples. The cause of this loss is not understood. There are considerable wall losses from the solid material (~ 50%). Wall losses from the PBL when generated alone are less than 15%. (This value includes vapor loss).

The concentration stability runs described above do not reflect very short term variations in the aerosol concentrations as determined immediately downstream of the generators. Both generators produce the aerosols in pulses, which result from the action of their respective feed systems. The PBL generator is fed by a piston pump which operates at 2 strokes/sec. The solid generator is fed by the screw feeder and material drops from the screw in discrete clumps, with the time between the drops dependent upon the rotational rate of the screw. In both cases, pulses of the aerosol are generated and are readily visible at the plenum. However, the concentration variations in the exposure chamber is minimal, as the volume of the chamber tends to dampen the pulses. The exposure chamber used for the developmental work is ca. 1.5 cubic meters in volume. A

PBL Aerosol Stability Test

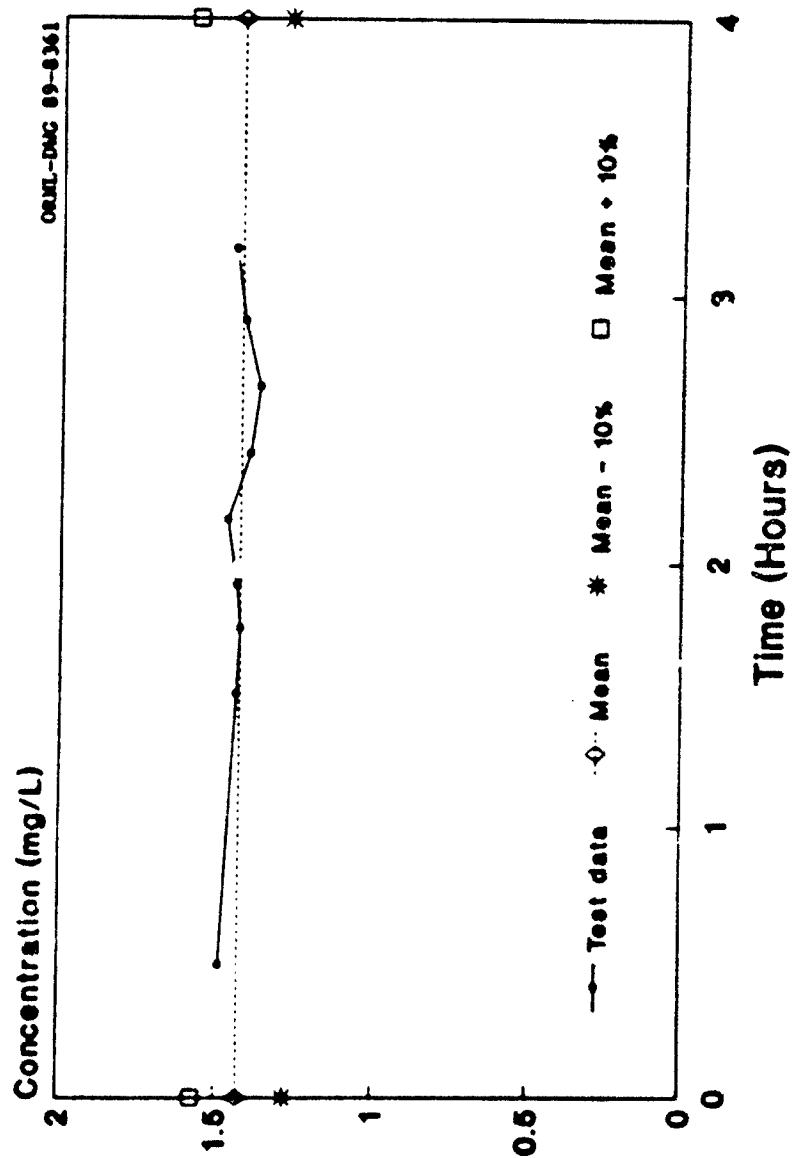


Figure 15. Stability Test; PBL Aerosol. PBL Aerosol Concentration vs. Time

Solids Aerosol Stability Test

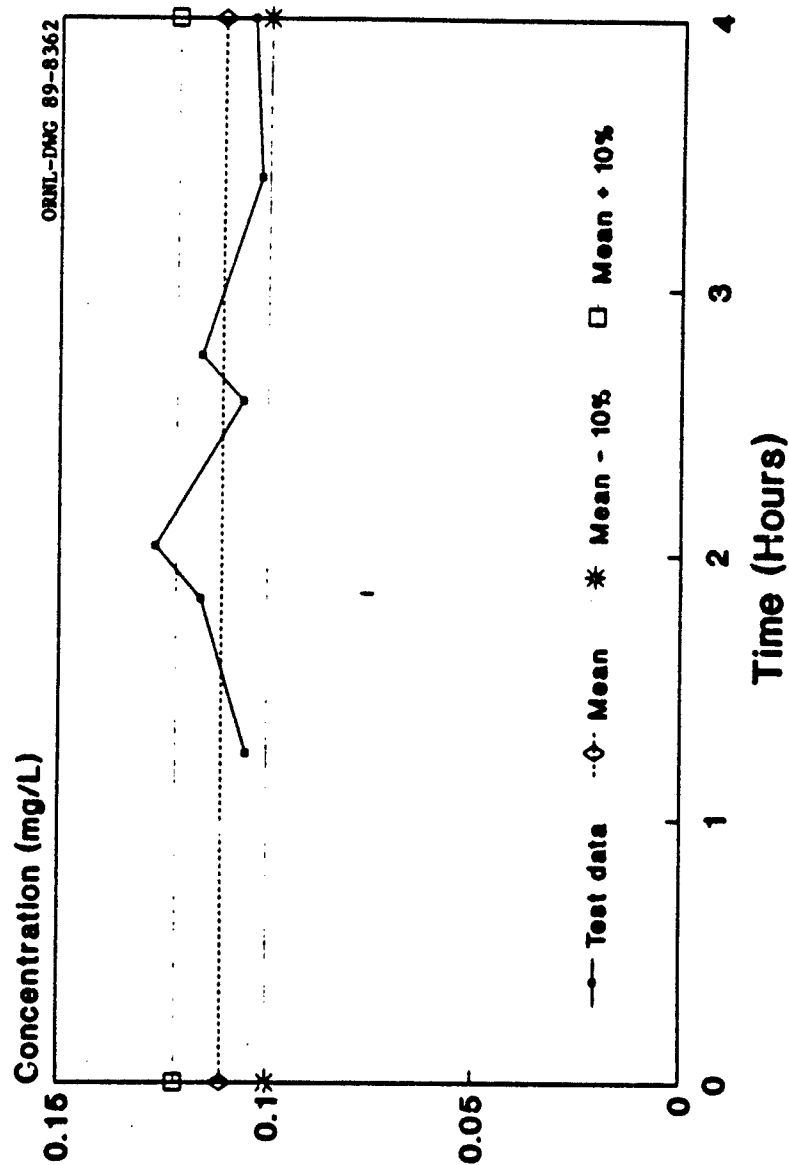


Figure 16. Stability Test; Solid Aerosol Concentration vs Time

Blended Aerosol Stability Test

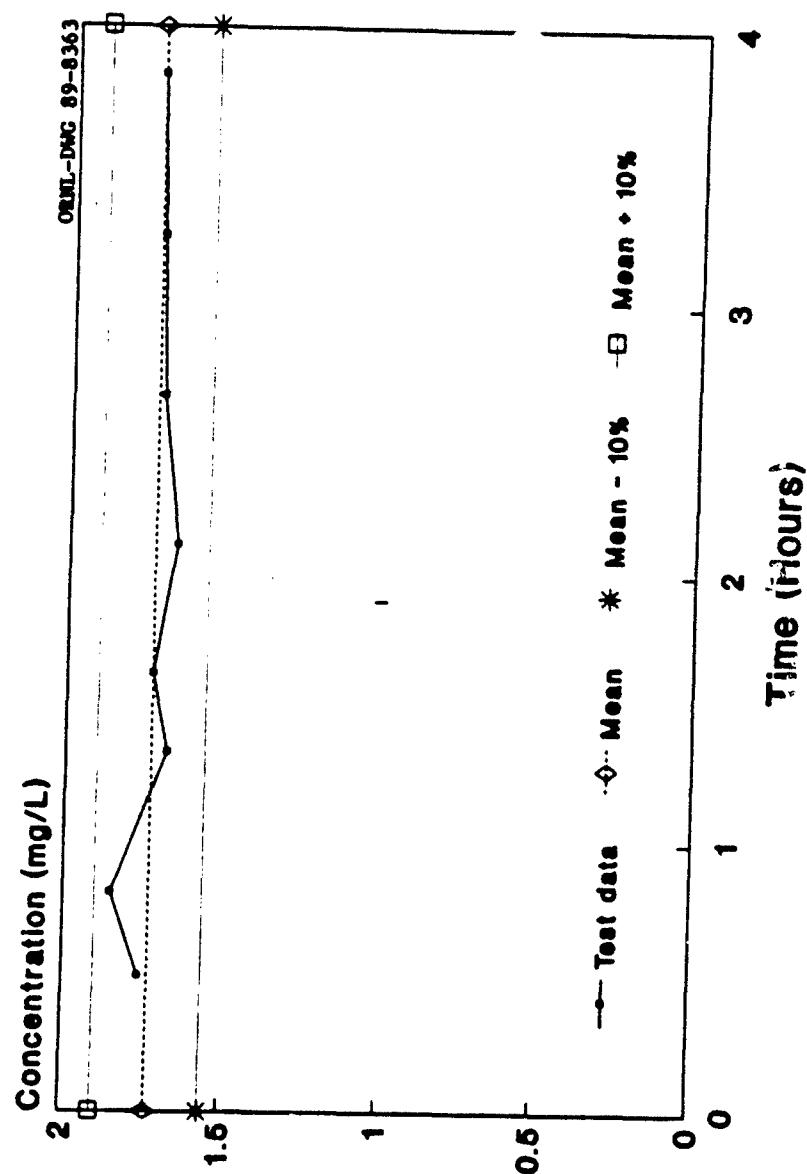


Figure 17. Stability Test; Blended Aerosols Blended Aerosol Concentration vs Time

flow rate of 500 liters/minute passes through the system. If the flow through the chamber was perfectly laminar, a given aerosol particle would pass through the chamber in about three minutes. Approximately 15 minutes is required for the chamber to reach equilibrium on filling or emptying. This means there is considerable turbulence, and incoming material will be mixed in the chamber.

Typical short term variations are shown in Figure 18. These data are from the same PBL run as presented in Figure 15. That run was also monitored using the ORNL light scattering aerosol monitor (Higgins et al, 1978). Note these data were obtained at discrete two minute intervals even though the monitor provides continuous readout of concentration. Although there are minor excursions from the average concentration (1.43 mg/L), the relative standard deviation of the central 51 points is slightly less than 3.5 %.

Chamber Temperature

The exit temperature of the PBL aerosol from the generator is near 700° F. With the dilution and cooling air added the temperature at the blending Y is typically somewhat greater than 90° F. The entrance to the 1.5 cubic meter chamber used for the developmental work is of the turret type and considerable cooling is achieved there. Temperature measurements within the chamber during extended runs indicated that the chamber atmosphere was only 3-4 degrees F. above ambient temperature.

PRECAUTIONS

A few precautions must be observed in the operation of the solid/PBL generating system in order for the system to function safely and reliably. Air flow for carrying and diluting the aerosol must be maintained without failure while the generator system is running. If animals are present the breathing air flow must be maintained even if the generator is not running. As previously stated, there is danger of reaching an explosive mixture of the PBL aerosol if dilution air flow is restricted. Flow monitors that provide low flow signals are available and should be installed in the exposure systems.

After the PBL generator is shut down, the PBL delivery tube from the pump to the generator should be disconnected at the generator input. Leaving the tube in place with fuel flow stopped has caused blockage of the delivery tube. This is apparently the result of pyrolysis of the heated fuel which remains in the tube. To remove the hot delivery tube, the operator must wear leather gloves for hand protection. Single thickness leather gloves are adequate.

Even with the addition of the vibrators and the stirring bar to the Accurate screw feeder, the solids in the hopper do not flow freely down the walls of the hopper. Periodically, the material must be dislodged from the walls. A reasonable depth of solid material in the hopper. (about 1/2 full over the stirring bar) must be maintained.

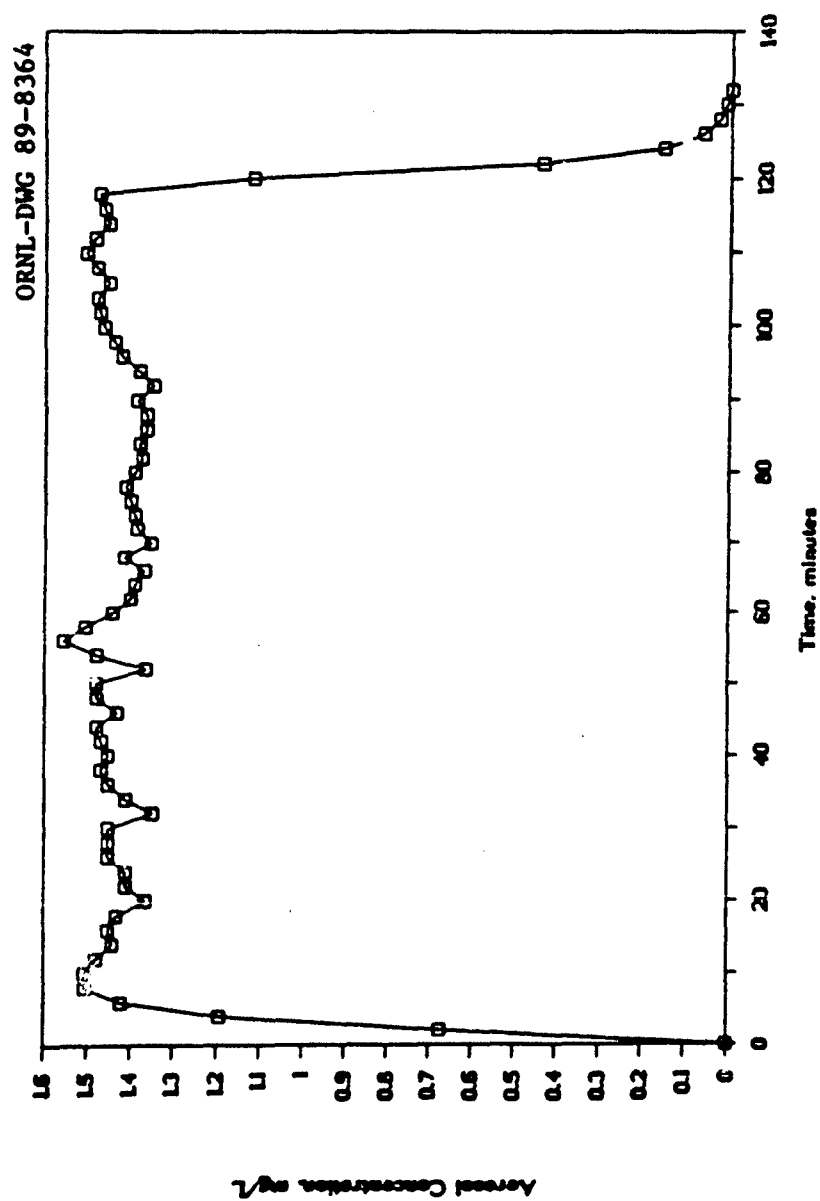


Figure 18. Stability Test; PBL Aerosol Using Light Scattering Technique
PBL Aerosol Concentration vs Time

The vibrators should be oiled daily. One drop of oil placed in the air line in front of the vibrator and blown through the unit seems to prevent blockage. During a run, the vibrator exhausts should be checked periodically to verify that they are operating. If they are occluded, a sharp rap is often sufficient to restart the vibrator action. The result of an inactive vibrator is a reduction of solids concentration in the aerosol mixture.

CONCLUSIONS

A system has been developed to generate a mixed aerosol of a Petroleum Based Liquid and a solid. The mixed aerosols are similar to that generated in a field unit and therefore should be applicable to inhalation toxicological testing. Limited chemical analysis of the PBL shows no significant chemical conversion during the aerosolization process. Particle size analysis reveals that the aerosols are well within the respirable range. The two generators show stability over time periods of a duration equivalent to daily animal exposure regimes. Exposure chamber temperature is only slightly above ambient. Some precautions for safe and reliable operation of the system are noted.

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APPENDIX

Component Descriptions and Drawings Petroleum Based Liquid Aerosol Generator

Generator tube

The generator tube is constructed of a 1-inch o.d. type 304 L stainless steel tube with two thermocouple ports, a nitrogen carrier gas port, and a fuel inlet port. The delivery tube is 3/8-inch type 304 L stainless steel. All joints for ports and the delivery tube are welded. The immersion heater is installed through a 1-inch compression tube fitting with a Teflon sleeve around the heater. See Figures A1 and A5

Immersion Heater

The heater is a 1000 watt, 120 VAC immersion heater (Figure A1) manufactured by Corning Glass Works (Cat. No. 16790). Heat is generated by a resistance heating element. The heater is incased in a Vycor sheath. The PBL is applied directly to the Vycor surface.

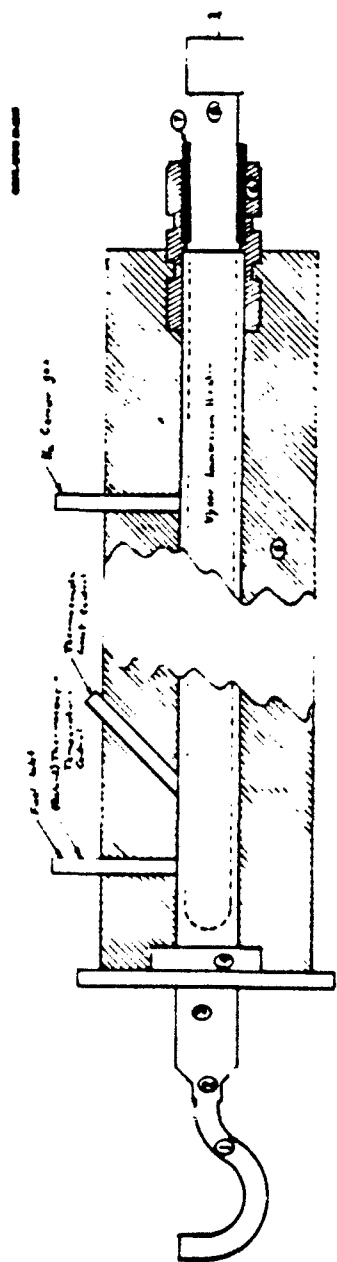
Flowmeter

A Matheson Model 7600 series flowmeter with a needle valve is used to meter the nitrogen carrier gas flow. The flowmeter contains the number 605 tube with both black glass and stainless steel floats. The nitrogen flow is normally set at 12 liters/minute.

Fuel pumping Assembly and Support

Figure A6 show the fuel pump assembly and support. The metering pump is an Eldex model E-120-S (Eldex Laboratories Incorporated). It is a single piston high pressure pump designed to deliver precise amounts of a liquid in a controlled manner. The pump rate is adjusted by changing the stroke of the piston by a manual micrometer. Pistons and check valve at inlet and outlet are sapphire. A 60 PSI pressure gauge (US Gauge Model 1530) is used to verify normal pump operation.

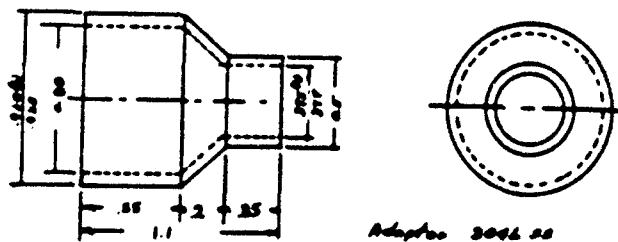
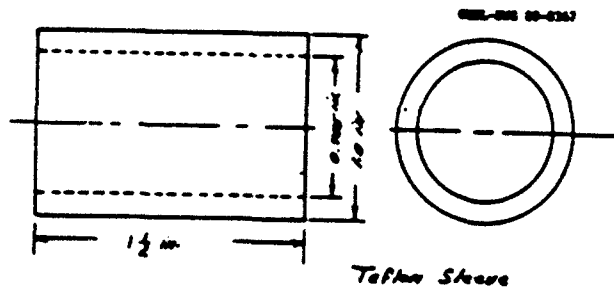
Just before entering the pump, the fuel is filtered through a Balston disposable filter (type DQ, Balston Inc.), positioned so as to not only remove solid degradation products from the fuel but also to trap air bubbles that might otherwise be introduced into the system. A ball valve (Whitey, SS41SX2) is used to direct the fuel flow to either the generator or to a vent position. The vent position is used during refueling to purge the pump of air bubbles. This operation is performed by drawing the fuel through the pump with a large syringe while operating the pump at high flow rate. The fuel reservoir is simply a polypropylene bottle with a compression type bulkhead fitting installed with teflon gaskets. All tubing is Teflon® or stainless steel.



Petroleum Based Liquid Rocket Generator Buy No. PBLG 1

Item	Quantity	Part No.	Notes
1	1	100-1000-1000	Upper Generator Nozzle
2	1	100-1000-1000	R. Generator Jet
3	1	100-1000-1000	Fuel Tube
4	1	100-1000-1000	Petroleum Based Liquid Rocket Generator
5	1	100-1000-1000	Upper Generator Nozzle
6	1	100-1000-1000	R. Generator Jet
7	1	100-1000-1000	Fuel Tube
8	1	100-1000-1000	Petroleum Based Liquid Rocket Generator
9	1	100-1000-1000	Upper Generator Nozzle
10	1	100-1000-1000	R. Generator Jet

Figure A-1 Schematic of PBL Generator Tube



Generator Diag. No. PBLG 3
(Sleeve)

00 to receive 1/2 in. tube
00 to pass inside 1 in. tube (optional)

Figure A-3 PBL Generator Teflon Sleeve and Adaptor

ORNL-DWG 89-8368

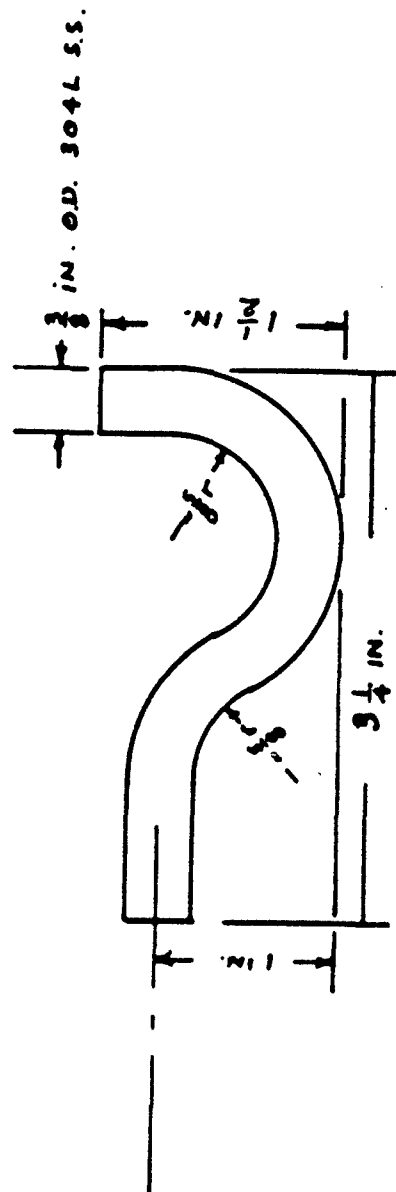


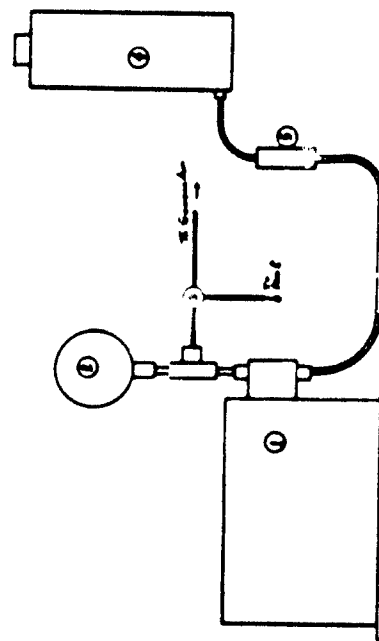
Figure A-4 PBL Generator; Delivery Tube

Technical drawing of a circular plate with the following specifications:

- Overall Diameter: $1\frac{1}{2}$ in.
- Mounting Holes: Four holes, each with a diameter of $\frac{3}{8}$ in. LD.
- Mounting Hole Spacing: $1\frac{1}{2}$ in. between centers.
- Central Hole: Diameter of $1\frac{1}{4}$ in.
- Side View: Shows a thickness of $\frac{1}{8}$ in.

Figure A-5 PBL Generator; Flange

Fuel Pump Assembly



0000-000-0000

Item	Model Number
1. Fuel Tank	MP 8130 S
2. Fuel Pump	Amelco 1000 S
3. Fuel Filter	Wichita 80 4000
4. Fuel Valve	Wichita 1000 S
5. Fuel Line	Wichita 8000 S
6. Fuel Nozzle	Wichita 8000 S
7. Pressure Gauge	Wichita 8000 S
8. Vent Line	Wichita 8000 S

Figure A-6 PBL Generator; Pump Assembly

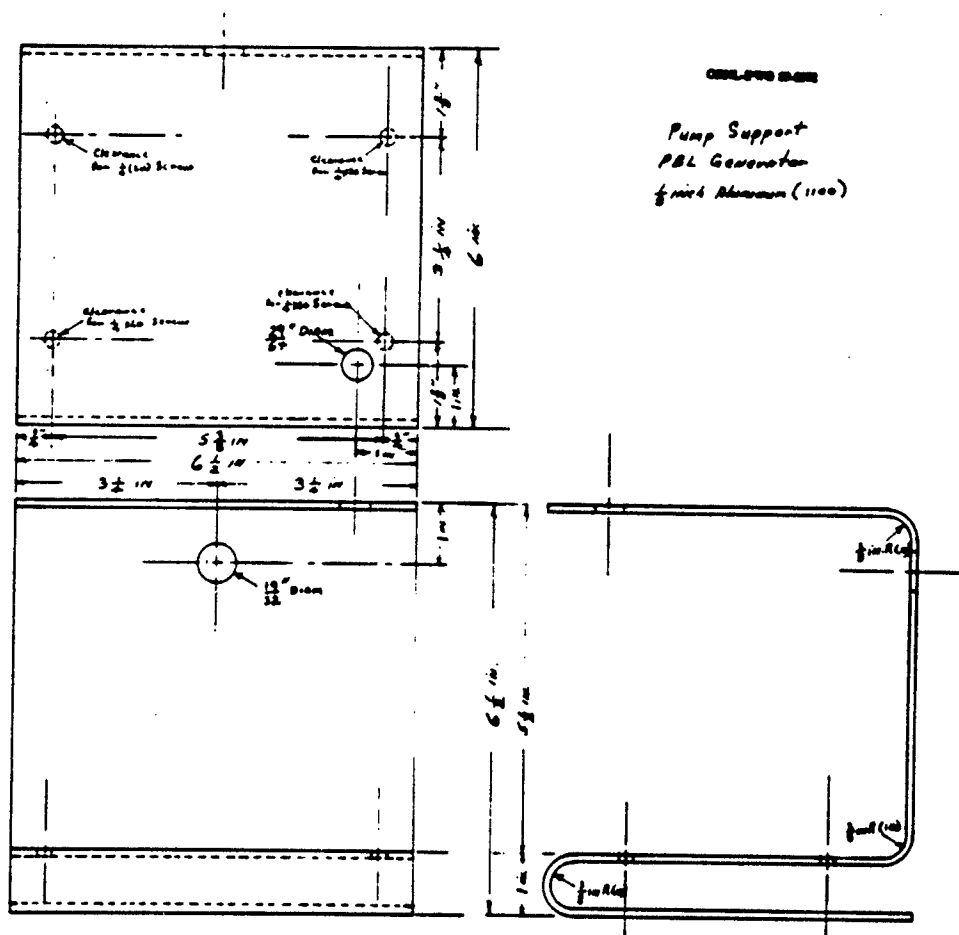


Figure A-7 PBL Generator; Fuel Pump Support

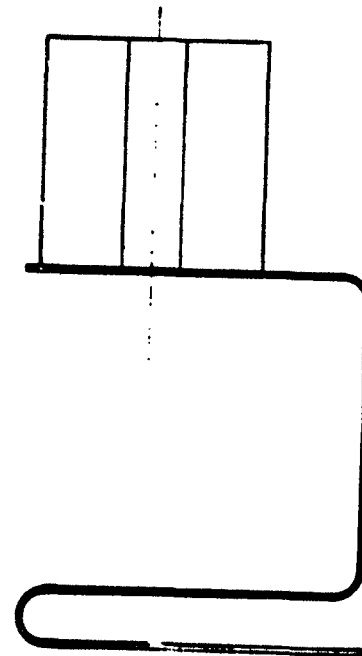
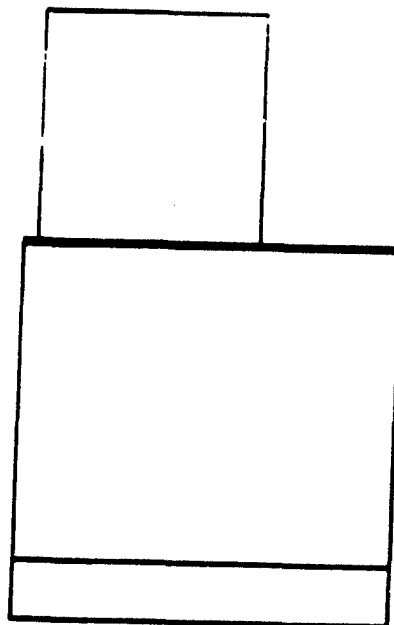
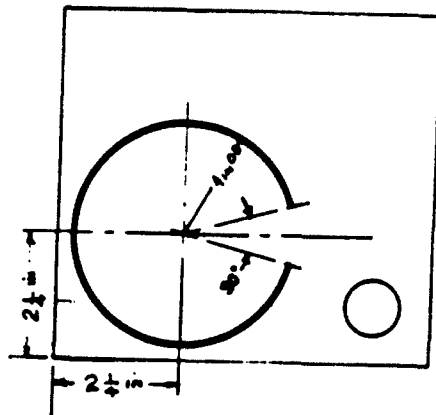
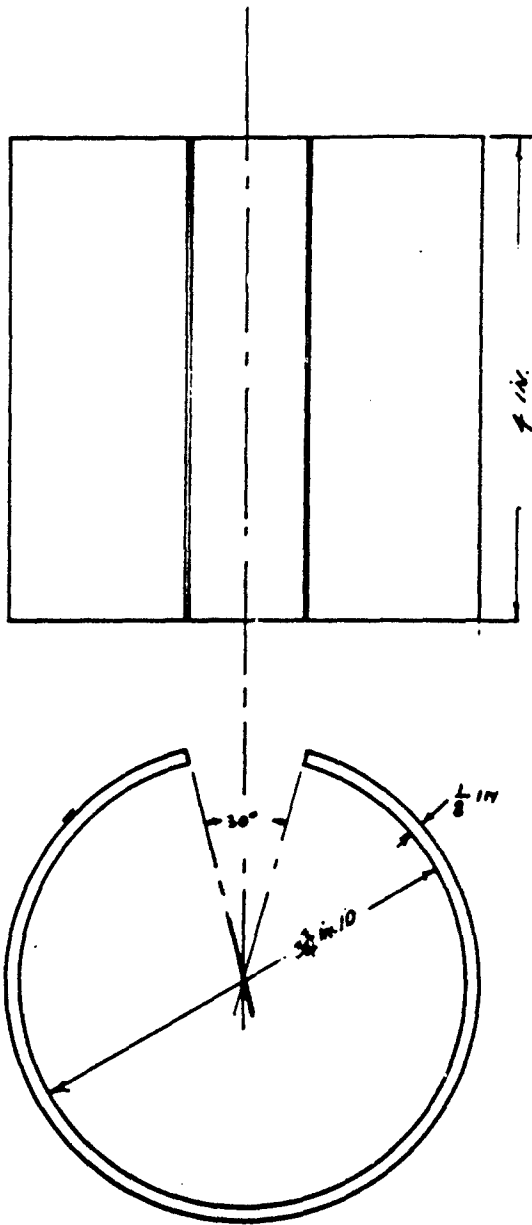


Figure A-8 PBL Generator; Fuel Pump Support

ORNL-DWG 89-8373



MATERIAL
 $\frac{1}{8}$ " 1100 Aluminum

PBL Generator
Fuel Reservoir Retainer

Figure A-9 PBL Generator; Fuel Reservoir Restraint

Flow rates from the metering pump range from 0.1 g/minute to just over 4 g/minute. At a flow rate of 500 liters/minute dilution air through the chamber, these delivery rates yield chamber concentrations of approximately 0.2 mg/liter to 8 mg/liter. The generator will supply sufficient material for animal exposures in chambers with a volume of about 1.5 cubic meter for whole body exposures, or for several branches of a nose-only exposure system.

Plenum

The plenum within which the PBL is mixed with the dilution/cooling air is a 2-inch diameter Pyrex flanged glass pipe tee. A two-foot section of flanged glass pipe is attached to the downstream arm of the plenum to permit some cooling of the PBL aerosol before passing into the flexible delivery tube.

Delivery tube

An 8-foot-long delivery tube connects the generator to a blending Y. The delivery tube is a 2-inch i.d. polyethylene flexible hose manufactured by the United States Plastic Corp., Lima, Ohio. The length of the delivery tube is effective in cooling the PBL aerosol to near ambient temperatures.

Temperature controller

The generator is operated at 600° C (1112° F). Chromel/Alumel type K thermocouples are used to monitor the temperature in the generator. The thermocouple is connected to a Barber Colman Model 527Z temperature controller capable of modulating up to 20 amperes into the controlled load. These controllers have digital set points (Fahrenheit scale), and analog deviation meters, and they provide proportional plus automatic reset control action. They also have an adjustable over-temperature alarm which is utilized for automatic shutdown.

A separate high temperature limit control (Barber Colman Model 121L) is incorporated in the system to provide an independent shutdown circuit, operating from its own thermocouple located near the Vycor heater. This is installed to prevent destruction of the generator should the Vycor heater control fail. One type of controller failure is known to result in uncontrolled heating of the generator. This failure was later traced to faulty contacts. Those contacts have been replaced with gold plated contacts and the problem has not reoccurred. The limit control protects against this failure. If either the controller or the limit control detects a temperature above a preset level, power to the network, including the heater, is shut off and can only be restored by manual reset after the high temperature condition clears.

A bypass switch is provided to override the automatic shutdown. This may be used for start-up, as, during initial heating, the system may overrun the set temperature sufficiently to activate shutdown. The bypass should only be used while the operator is

present and observing the system. For normal operation the bypass should be locked off. A key lock is provided.

Three auxiliary 110-VAC outlets are provided in the controller. It is recommended that the fuel metering pump be supplied from one of these outlets. If a high temperature excursion is detected and shutdown activated, the metering pump will also be stopped, preventing flooding of the generator with oil. The remaining outlets may be used for visual or audio alarms. Circuit diagrams for the entire control system are provided separately.

Housing

The heating element of the PBL generator is controlled at a temperature of 600° C. External surfaces, particularly the mounting bracket become very hot. To protect the operator from contacting these surfaces, the system has been mounted in an aluminum case with an access door. (Portable Aluminum Instrument Case TC 304 GT, Bud Industries)

Solids Dispersal System

Jet Mill

The system that de-agglomerates the solids is a jet mill (Jet-O-Mizer Model 00, Fluid Energy Processing and Equipment Company) within which the particles are accelerated to high velocities in the grinding chamber by two jets of air. The solids are swept into a turbulent motion in which they collide and pulverize one another. The particles are carried into a classifier zone where the small particles leave the mill in the outlet flow. Large particles are returned to the mill grinding or de-agglomeration zone where they are further reduced and returned to the classifier. The jet mill was modified by the addition of the extension of the delivery tube and the shroud on the funnel. See Figures A10 and A11.

Screw Feeder

The delivery rate of the solids is controlled by a screw feeder that supplies the jet mill. The screw feeder (Series 100 by AccuRate Inc.) is fitted with a vinyl hopper that is flexed by a massaging paddle that helps to prevent bridging of the solids over the screw. The screw carries the material from the hopper through a delivery tube, dropping the material into the inlet funnel of the Jet-O-Mizer, where air aspiration pulls the solids into the Jet mill. Figure A10 is a side view of the solid aerosol generator showing the Jet-mill dispersal unit with the screw feeder in place. Modifications to the screw feeder include the use of a stirring bar and two air driven vibrators (Model VM-25, Cleveland Vibrator Company). One vibrator is attached to the stirring bar while the second is attached to the delivery tube. See Figures A10 and A11.

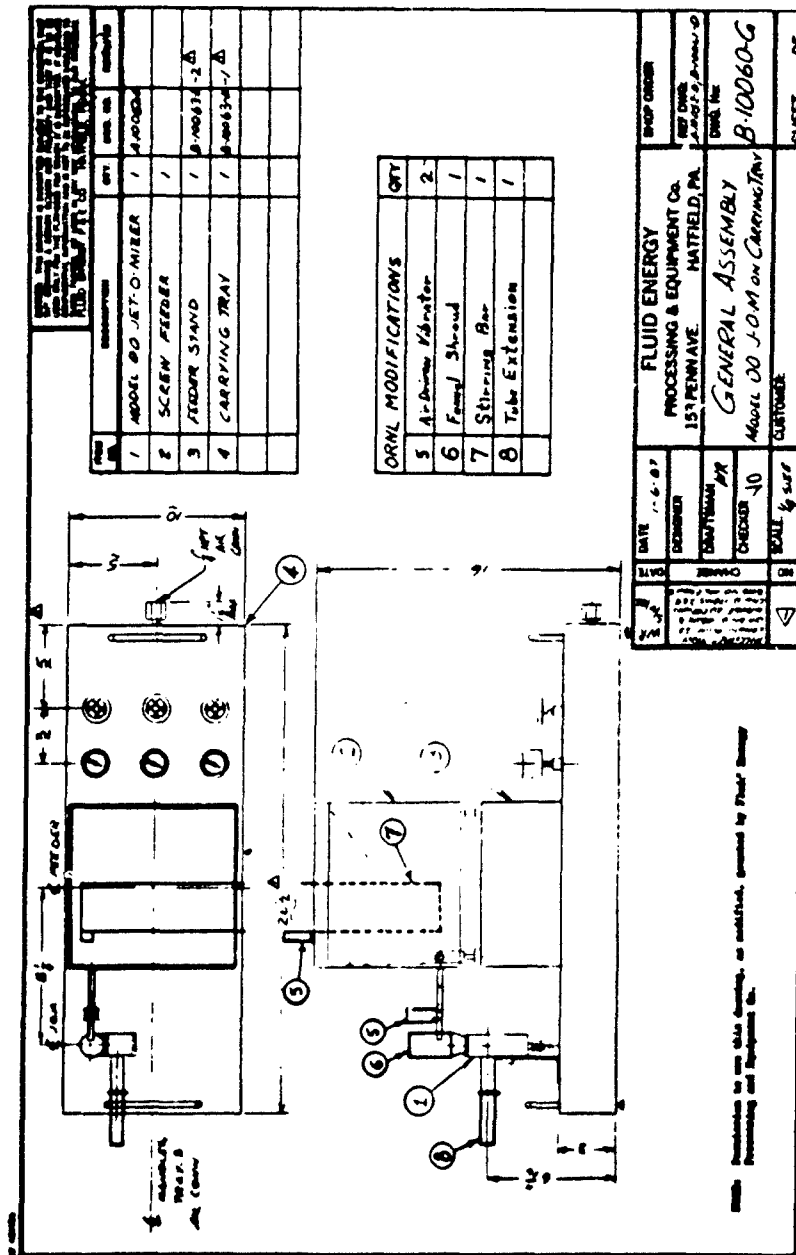


Figure A-10 Solids Aerosol Generator; Feeder and Jet Mill with Modifications

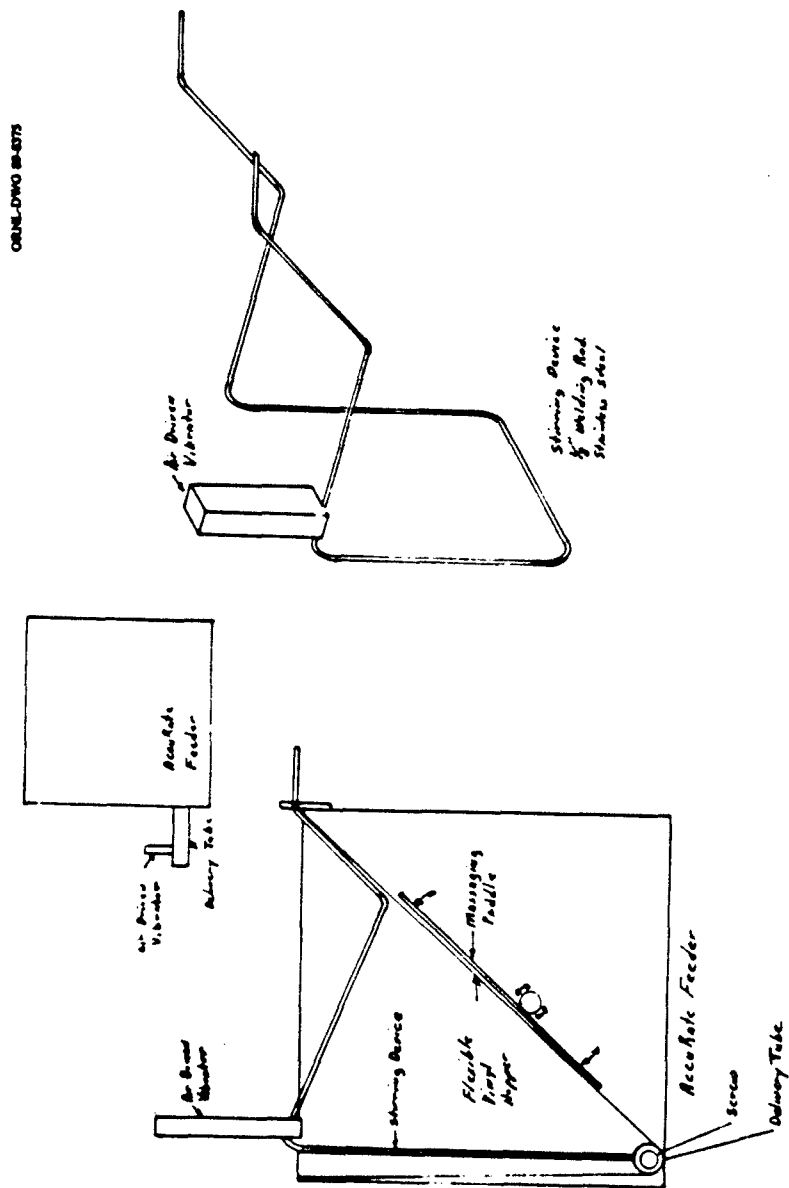


Figure A-11 Solids Aerosol Generator; Stirring Bar and Vibrator Modifications

Plenum

The plenum for the solids dispersal system is a 2-inch glass pipe tee modified by the addition of two tubes. One of these tubes, mounted in the vertical position, serves to collect particles impinged against the plenum wall upon exiting the Jet-O-Mizer. The tubes also permit the operator to remove these collected materials without dismantling the plenum. See Figure A12.

Housing

Housing for the Jet-O-Mizer and the screw feeder is a 1/2 inch plywood box that serves both to reduce noise levels to the operator and to confine fugitive solid material. The housing is painted with an enamel paint to provide a smooth surface that may be easily cleaned. The top to the housing is of clear Lucite so that operations of the screw feeder may be observed without opening the container. See figure A13.

Blending Y

The two aerosols are carried from the generator through the flexible 2-inch hose and are blended at a Y before being delivered to the exposure chamber. The developmental system employs a stainless steel Y. However, a polyvinyl chloride Y commonly used in plumbing has been incorporated for the latter models.

Utility Requirements

Petroleum Based Liquid Generator

Electrical

The PBL generator requires 110-VAC power to the temperature controller. The heating element is a 1000-watt unit. The metering pump is rated at 10 amperes.

Nitrogen Carrier Gas

The carrier gas through the generator is nitrogen, at a flow rate of ca. 12 liters/minute. This is a significant demand for nitrogen, and one conventionally sized cylinder will last for only approximately 4 hours. It is recommended that if several generators are to be operated simultaneously a liquid nitrogen container equipped to deliver nitrogen gas be installed for the nitrogen supply. A cryogenic system manufactured by Union Carbide (POL Model PGS-45) has been used with similar generators for diesel fuel aerosol to supply four generators but at somewhat reduced flow rate. Only 10 liters/minute nitrogen flow was required for that generator. The nitrogen system was sufficient to supply four generators operating simultaneously for up to 6 hours per day for 5 days/week. The cylinder was refilled on a weekly basis. (Holmberg, et al, 1982)

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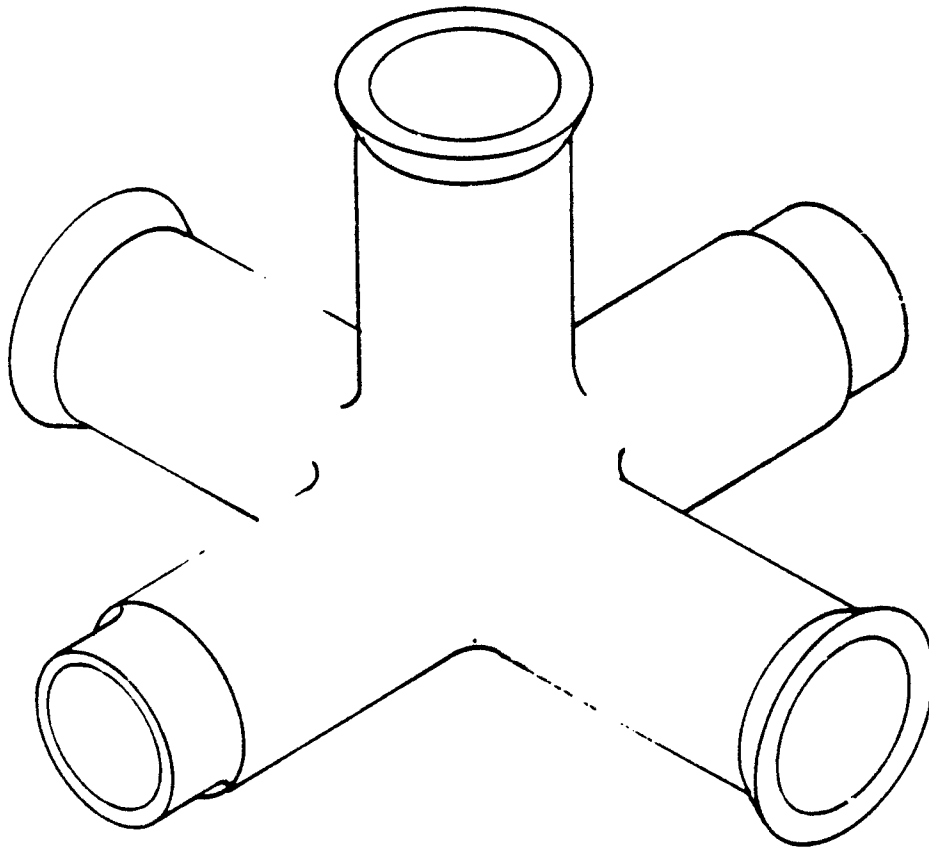


Figure A-12 Solids Aerosol Generator; Plenum

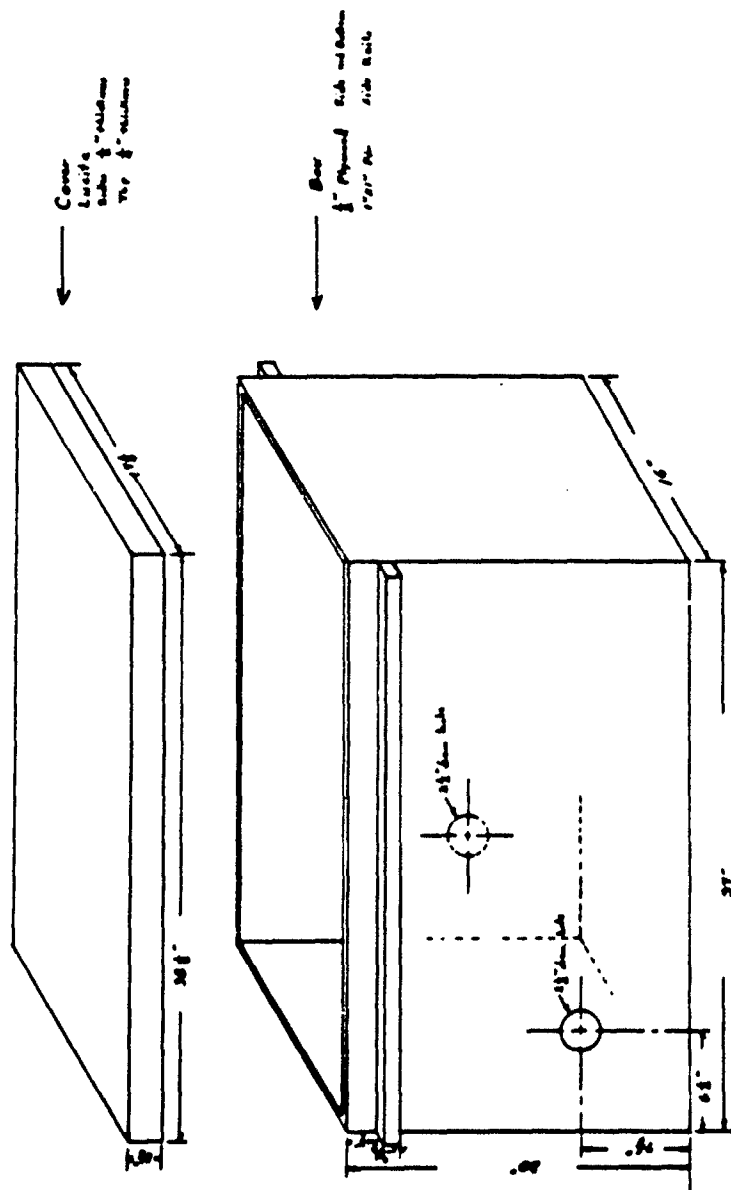


Figure A-13 Solids Aerosol Generator; Housing

Solid Dispersal Unit

Electrical

The screw Feeder operates from 110 VAC and is fused at 1 amp.

Air Supply

The air supply to the two air-driven vibrators is 15 PSI, (The manufacturer recommends 30 PSI air. The vibrators were found to operate satisfactorily from house air nominally 15 psi).

The Jet-O-Mizer is designed to operate on compressed air up to 110 psi. Three valves meter the air to the aspirator jet and the two de-agglomeration jets of the Jet-O-Mizer. The air supply used for the developmental system, nominally 100 PSI, would routinely drop to 90 to 95 PSI after all three valves of the Jet-O-Mizer were opened.

Installation and Operating Instructions PBL/Solid Aerosol Generating System

Installation of the PBL Generator

Place the PBL generator on a solid surface. Only minimal heat will be radiated through the box to the surface under normal operation, but a wooden surface should be avoided unless protected by a heat shield.

Install the plenum (2-inch glass pipe tee) with the side arm over the delivery tube of the generator and the run of the tee in a vertical position. The bottom opening of the run should be free of obstructions, since this is the dilution air inlet.

Attach the 2-foot section of 2-inch glass pipe to the upper end of the tee with the pipe flange provided. As the transfer tube connects to the swaged end of this glass pipe, some support for this tube will be required.

Connect the 2-inch flexible hose to the swaged end of the glass pipe delivery tube and anchor with the hose clamp provided.

Connect the blending Y to the inlet of the exposure chamber and then connect the downstream end of the flexible delivery tube to the Y. Again anchor the flexible hose in place with a hose clamp.

The rearmost 1/4-inch port in the generator is the nitrogen carrier port. Connect the nitrogen port to a nitrogen gas source through the rotameter.

Adjust the nitrogen gas pressure to 16 psi. Using the metering valve of the rotameter, set the nitrogen flow at 12 L/minute at 16 psi.

Connect the generator heater power cord to the temperature controller through the outlet marked "CONTROL", with one of the power cords provided. A second power cord, also provided, connects the temperature controller to a 115-VAC outlet.

The system is delivered with the two thermocouples in position. To replace a failed thermocouple, loosen the Parker fitting nut and remove the faulty thermocouple. The Vespel ferrule holding the thermocouple in place is drilled to fit the thermocouple and may be frozen in place, and force may be needed to remove it from the thermocouple. The control thermocouple, mounted in the generator, adjacent to the fuel delivery tube, should be mounted so as to be at the surface of the immersion heater but not forced against the surface. Tightening the retaining nut can pull the thermocouple against the heater wall with great pressure and could break the Vycor sheath. The high temperature shutdown thermocouple is mounted through the port just behind the fuel port and the control thermocouple port. This thermocouple is mounted so as to lie along the surface of the immersion heater in the heating zone and is held in position by a vespel ferrule in a Parker fitting.

Connect the control thermocouple (The control thermocouple is mounted beside the fuel delivery tube) to the thermocouple connector marked "CONTROL" at the left side of the temperature control system.

Connect the high temperature shut down thermocouple to the thermocouple connector marked "LIMIT". (The high temperature shut down thermocouple is mounted to the rear of the fuel delivery tube).

The PBL fuel delivery tube (1/16-inch stainless steel tube connected to the metering pump through a 1/8-inch Teflon® tube) is inserted through a 1/4-inch tube near the downstream end of the generator. Insert the tube through the 1/8" to 1/4" tube reducing union until the tip of the delivery tube just touches the surface of the heater. Back the delivery tube away from the heating tube surface until it breaks contact with the surface, and tighten the swage nut. Again, use care to not force the delivery tube into the wall of the heating element. Once the tube has been swaged into position, the ferrule should remain in position for future placement.

We have experienced some plugging of delivery tubes if they are left in place with no oil being pumped. The tube should be inserted just before starting the aerosol generation and removed at the end of the generation period. A pair of leather gloves (single thickness) is sufficient to protect the hands from heat conducted to the Parker fitting retaining the delivery tube. It is best to use a swage-type cap to seal the access port after removing the delivery tube.

Operation of the PBL Generator.

Before operating the PBL generator, the nitrogen flowmeter and the PBL metering pump should be calibrated. The metering pump flow is regulated by length of piston travel which is in turn adjusted with a micrometer. Calibration of the metering pump is best performed by weighing timed deliveries of PBL at preset micrometer positions. The calibration is nearly linear over the range of the pump.

1. Turn on the nitrogen flow and check to verify a flow rate of 12 L/min.
2. Set the temperature controller to 1111° F (600° C) on the digital set point on the front panel.
3. Set the limit control to approximately 1200° F. To set the temperature, insert the tool provided through the hole in the free turning knob and engage the pointer set. This procedure prevents accidental change of the set point.
4. Move the power switch to the "ON" position, and depress the reset switch momentarily. The heater should begin to heat and the power light (this light is on when voltage is applied to the controlled heater) will be lit. After a short waiting period, the analog gauge will display the temperature. The range of the gauge is approximately 200 degrees above and below the set point. As the temperature approaches the set point (gauge midpoint), the power light will begin to flash. As the temperature passes the set point the power light will go off and no voltage will be applied to the heater. During initial warm up, the temperature will override the set point and then cool. As the temperature reaches the set point from the high side, the power light will begin to alternately turn on and off as voltage is intermittently applied to the heater. The temperature should stabilize quickly. Normally the system is permitted to equilibrate for approximately 15 minutes to insure that heat is distributed uniformly through the system.
5. With the fuel delivery tube disconnected from the generator, fill the PBL reservoir with fresh PBL. Note that PBL on standing while exposed to air and light will degrade, darkening and forming some sediment. For this reason the fuel is filtered between the reservoir and the pump. After filling the reservoir, turn the switching valve to the vent position and turn the pump power switch on. Adjust the micrometer to approximately the 250 value. Start the pump and while it is running use a small syringe to pull the PBL through the pump. This process effectively pulls air bubbles through the check valves and the delivery lines. After the air has been removed from the system, move the switching valve to the run position and permit the delivery line to fill with the PBL. The delivery tube should be placed in the vent hole in the fuel reservoir for this operation. Turn off the pump power

switch and install the delivery tube in the fuel port of the generator. Note that if the delivery tube and line is positioned below the fuel reservoir, fuel can feed by gravity through the pump and the delivery tube. The fuel pumping assembly should always be mounted below the generator. When not in the generator, place the delivery tube in the hole in the fuel reservoir top.

6. Set the metering pump micrometer for the selected delivery rate based upon the pump calibration and the air flow rate (dilution air) through the exposure chamber. For example, if 1 mg/L of PBL aerosol concentration is required, and the total chamber air flow is 500 L/min., then 500 mg/minute of PBL must be pumped through the generator. Due to flow measurement errors, pump calibration errors, and loss of volatile components, the delivery of aerosol will not be exactly that predicted from the pump rate (usually 10-15% lower). Concentration must be monitored (verified) by filtering and weighing a sample of the aerosol. After the system has been calibrated and some experience gained in its operation, the concentration settings can be set rather closely.
7. After satisfactory warm up of the generator (-15 min.).
 - a. Verify nitrogen flow through the generator (12 L/min.)
 - b. Verify that cooling and dilution air through the generator plenum to the chamber is at the proper level. This flow rate is usually 500 L/min. if operating the PBL generator only. When operating the PBL and solid generator together, the larger fraction of the air, ~300 L/min., passes through the solid generator plenum while ~200 L/min passes through the PBL generator plenum. Final dilution of both aerosols upon blending is from both air streams as they are blended at the blending Y.
 - c. Activate the metering pump to deliver the PBL to the heated zone of the generator. The aerosol should be immediately visible in the plenum and then in the exposure chamber. Permit the generator to run for 10 to 15 min. and check the concentration by filtration. Depending upon chamber size, air flow rate and mixing, it takes approximately 10 to 15 minutes for the concentration of aerosol to reach equilibrium. Samples should be drawn periodically during a run to verify concentration.

Shutdown

To stop generation of the aerosol

- a. Turn the switching valve of the pump system to the vent position.
- b. Remove the delivery tube from the generator and place the delivery tube in the hole of the reservoir cap.

- c. Cap the fuel port.
- d. Turn off the pump power switch.
- e. If further runs are not required, turn off the generator heater system but continue to pass the nitrogen carrier gas through the generator until it cools.
- f. After the generator has cooled, (approximately 15 minutes), turn off the nitrogen carrier.

Changing the fuel filter

The filter in the fuel line is to protect the check valves of the metering pump and prevent clogging. It also serves to collect air bubbles introduced into the system. This filter should be changed periodically. Carbonaceous material will collect in the filter and will eventually clog it. To replace, simply loosen the two 1/4-inch Parker fittings retaining the filter. Teflon ferrules are used and the filter should slide easily out of the fitting. Replace it with a new filter, observing the directional arrow on the filter, and tighten the two fittings. After replacing a filter, remove air from the filter using the syringe technique as described previously.

Precautions

As a precaution, ground the generator tube to a suitable ground. This should prevent electrical shock in case of breakage of the Vycor heater sheath, exposing the heating coil.

Certain parts of the generator become hot enough to cause serious burns. Attempts have been made to shield the operator from these surfaces. However when working with the hot generator, care should be taken to avoid contact with these surfaces, and, if contact must be made, wear suitable leather gloves to protect the hands.

Avoid operating the heater unless carrier gas is being passed through the generator. The carrier promotes even heating throughout the generator and prevents hot spots from developing. Enough heat can be generated with the heater to melt the stainless steel tube if uncontrolled heating occurs. This is not likely to occur with the present temperature control.

Installation, Solid Aerosol Generator

Connect the glass pipe plenum over the delivery tube using the foam plastic sleeve provided. Anchor the glass plenum in position with the two swaged outlets horizontally so as to connect with the flexible delivery tube. The tip of the generator delivery tube should be very close to the wall of the plenum (-1/8 inch). Anchor the plenum in this position using the clamp provided, connecting the utility clamp to the forward carrying handle of the Jet-O-Mizer.

Place the Jet-O-Mizer in the wooden housing with the delivery tube to the end of the housing with the 2 1/2-inch diameter holes in each side of the housing. These holes are provided for the flexible delivery tubing.

Connect the 100-psi air line to the 1/4-inch diameter stainless steel tube mounted through the rear wall of the housing and connect that SS tube to the inlet of the Jet-O-Mizer.

Place the desired screw or helix in the AccuRate screw feeder. Place the screw feeder on the stand of the Jet-O-Mizer. The delivery tube from the screw feeder should pass through the hole of the shroud around the Jet-O-Mizer funnel and be so positioned that the solid material will drop near the center of the funnel.

Connect the two clear PVC lines to the inlet ports (bottom port) of the two vibrators. One vibrator is mounted on the stirring bar and the second is mounted on the delivery tube of the feeder.

Connect the outlet port (upper port) of the vibrators to the two PVC tubes passing through the housing wall. These tubes direct the spent air from the vibrators to the outside of the housing and prevents turbulent air from dispersing the solids in the housing.

Connect the vibrator compressed air feed line to a 15 psi air source.

Pass the aerosol delivery tube (2" id flexible hose) through one of the holes in housing wall and connect the hose to the plenum. Connect the other end of the hose to the blending Y affixed to the inlet of the exposure chamber. Seal the flexible hose in the housing wall with Silastic.

Another section of the flexible hose should be connected to the opposite swaged arm of the plenum through the wall of the housing. This tube is effective in reducing the noise of the generator in the vicinity of the operator. By positioning this inlet tube to a site away from the generator, much of the high frequency sound is muffled.

Direction of the air flow through the plenum is optional and depends upon user arrangement.

Operating the Solids Aerosol Generator

General operating manuals for the screw feeder and the Jet -O-Mizer are provided. The following instructions apply in operating the modified systems as assembled for this aerosol generator.

1. Set the potentiometer of the feeder to produce the desired feed rate. The concentration of the solid aerosol will depend upon the feed rate (which in turn depends upon the rotation rate of the screw, the size and pitch of the screw) and the dilution air flow rate. Calibration of the feeder delivery rate as a function of the rotation rate of the screws are in figures A-14 - A-16. A calibration of the turn rate (RPM of the screw vs the potentiometer setting is in the Appendix (Figure A-17). Aerosol delivery will be only approximately 50% of the concentration based upon feed rate and dilution air rate. The losses are chiefly impingement losses in the plenum and wall losses to the delivery tube and chamber. These losses will vary with the exposure and delivery system used and calibration for each unit will be necessary.
2. Fill the hopper of the feeder with the solid material.
3. Verify the dilution air flow rate through the plenum of the generator.
4. Turn on the 15-psi air to the vibrators and verify that they are operating.
5. Turn on the 100-psi air to the generator.
6. Start the solid aerosol generation by starting the feeder. If the screw is not filled with the solids, there will be a delay in delivery of the solid aerosol until material feeds through the screw and into the inlet funnel of the jet mill. Generation should continue smoothly, but an operator should periodically check the system, particularly the following:
 - a. Observe the feeder hopper. Occasionally the solid material does not feed down from the walls of the hopper smoothly and this can affect the delivery rate if material is depleted over the screw. Stirring the material manually from the walls is occasionally necessary. Maintain a sufficient supply of solid in the hopper, a minimum of two to three inches in depth over the screw.
 - b. Observe the vibrators. These have jammed on occasion. Daily oiling by placing a drop of light machine oil in the air supply line just in front of the vibrator is sufficient to maintain lubrication.
 - c. Maintain a check of the dilution air flow through the system. The concentration of the aerosol is directly related to the dilution air flow. Loss in air flow may indicate difficulties with the spent aerosol removal system. Pressure drops measurements across the removal system are helpful.

7. After the system has operated for approximately 15 minutes, verify the chamber concentration by collecting a filter sample of the aerosol.

Shutdown

To avoid material being fed into the funnel of the Jet-O-Mizer which is not moved through the mill, shut down should be in the order as follows:

1. Turn off the screw feeder.
2. Turn off the air to the vibrators.
3. Turn off the 100 PSI air to the Jet-O-Mizer.
4. Dilution air should be drawn through the exposure chamber for at least 15 minutes before opening the chamber door. Even after the aerosol has been removed from the chamber, care should be exercised in opening the chamber door. The walls of the chamber will be thoroughly coated with the aerosol material and if the door is opened so as to create turbulent air, much of the material may be dislodged into the exposure room. The material, is quite slippery and in sufficient quantity it can create a significant walking hazard. All spills, whether of the solid, PBL or mixed materials should be cleaned up immediately. If cleanup is delayed, the area should be marked off to prevent persons unaware of the hazard from entering.

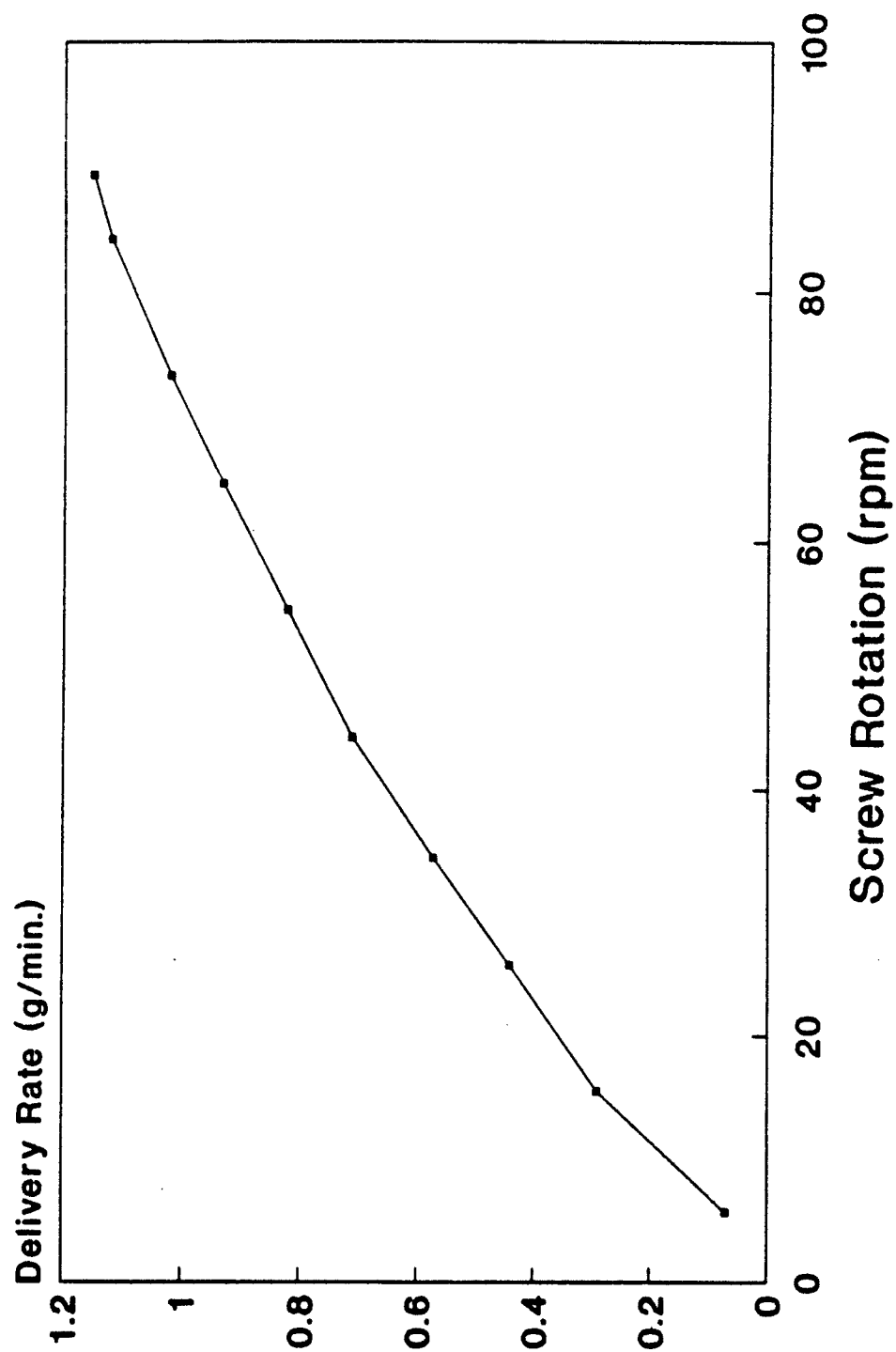


Figure A-14 Solids Aerosol Generator
Accurate Feeder Calibration (1/4 inch Helix)

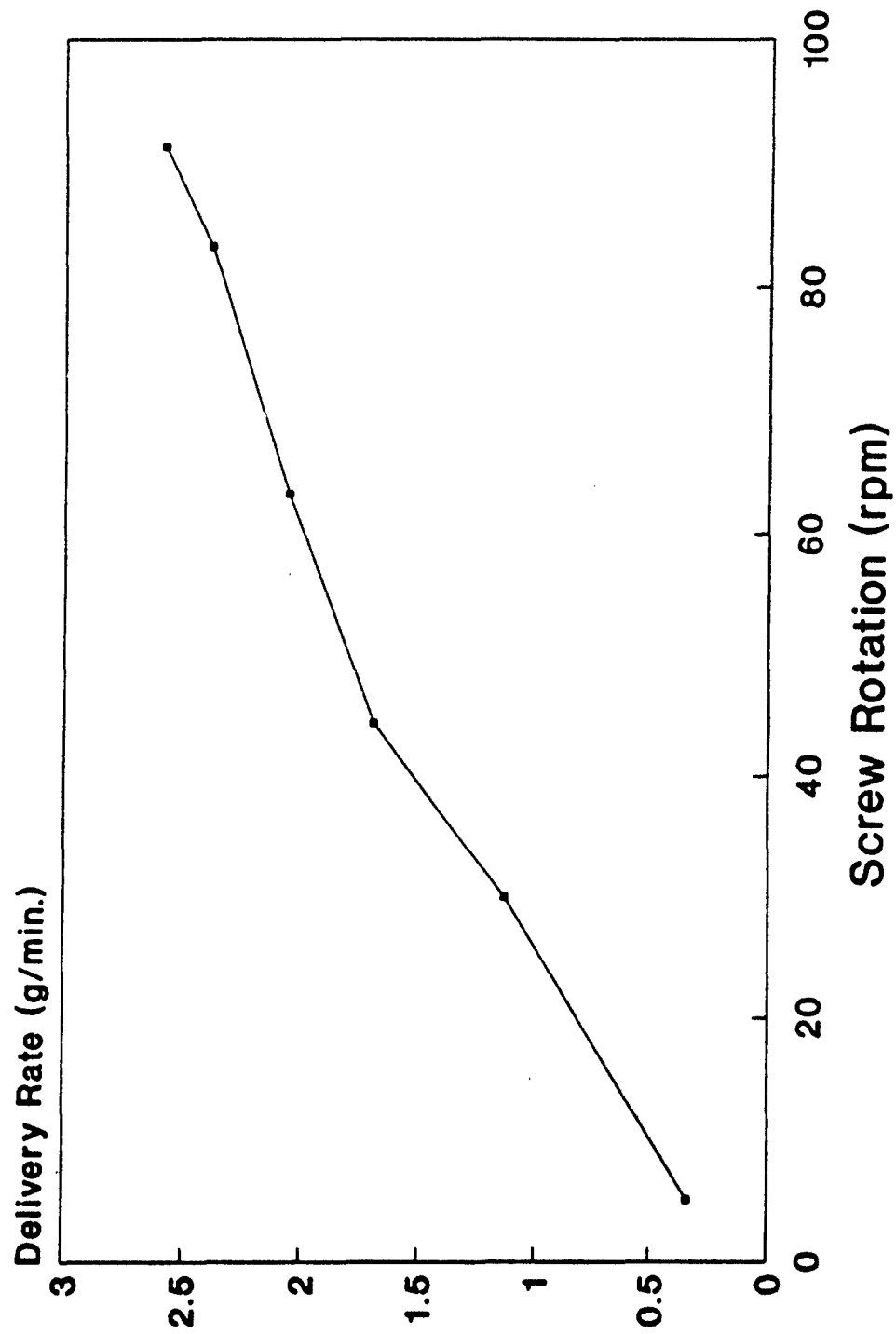


Figure A-15 Solids Aerosol Generator
Accurate Feeder Calibration (3/8 inch Helix)

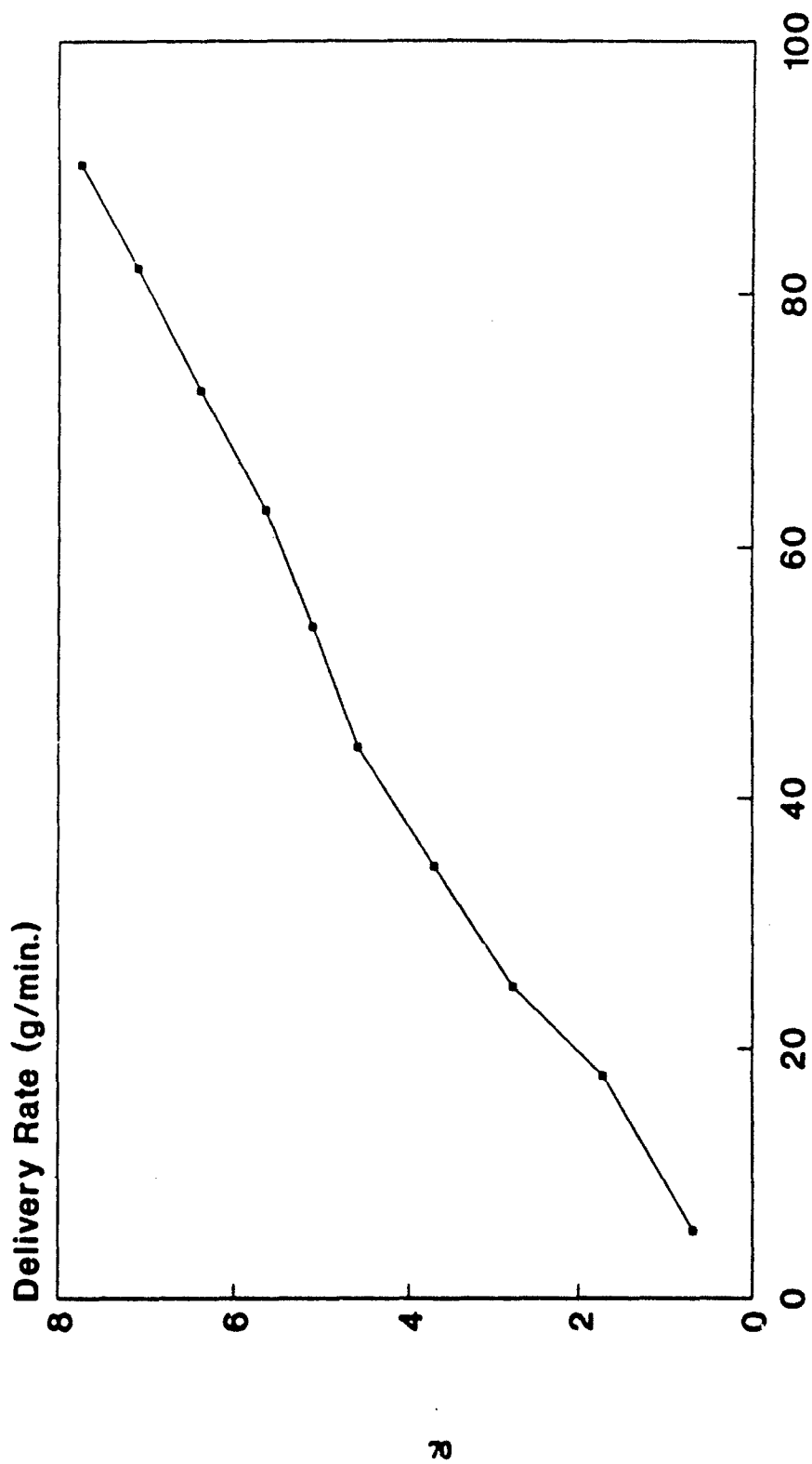


Figure A-16 Solids Aerosol Generator
Accurate Feeder Calibration (1/2 inch Helix)

RPM vs Potentiometer Setting

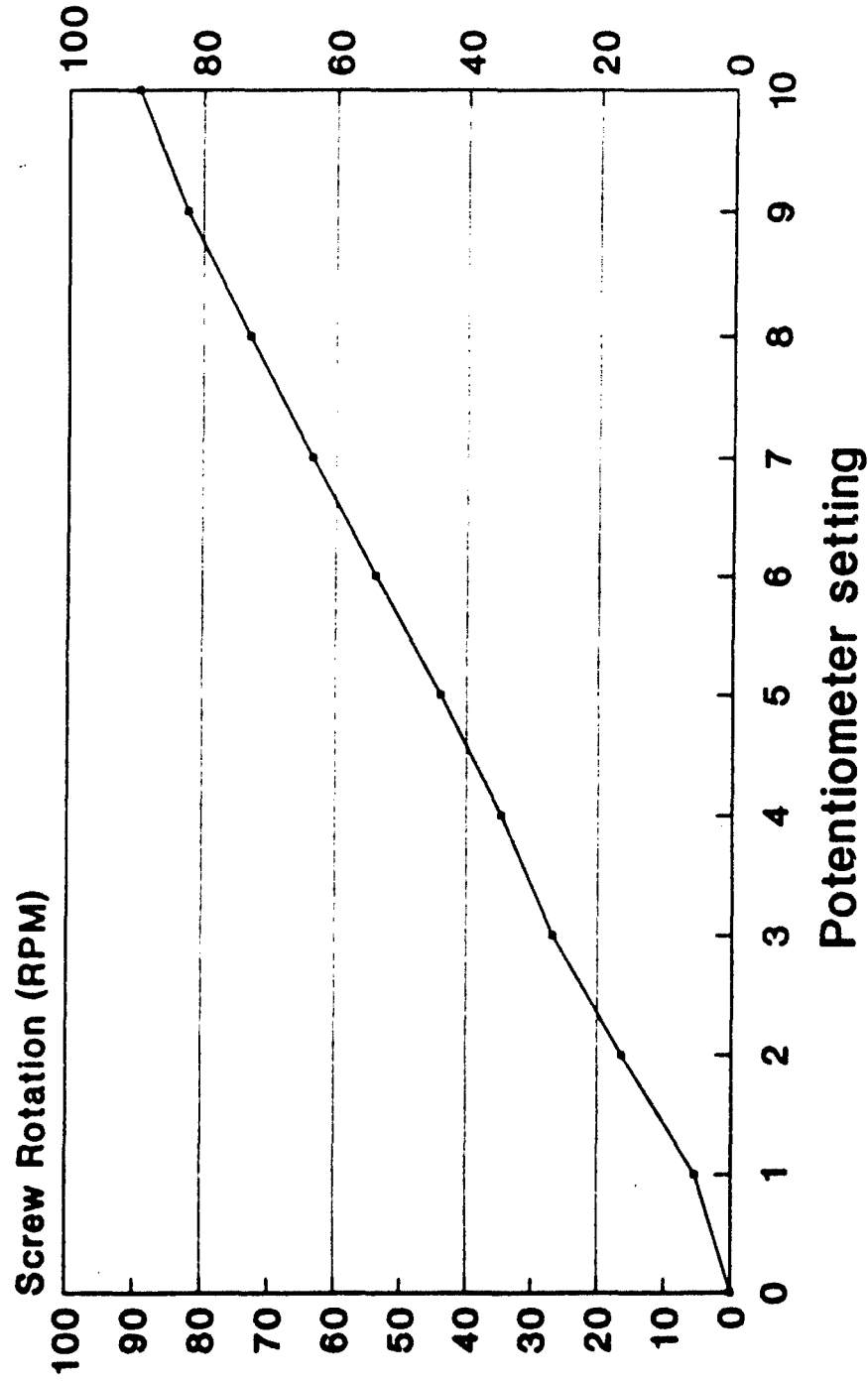


Figure A-17 Feeder Screw Rotation Rate Calibration

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